Los Alamos Coated Conductor Development

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Superconductivity Technology Center
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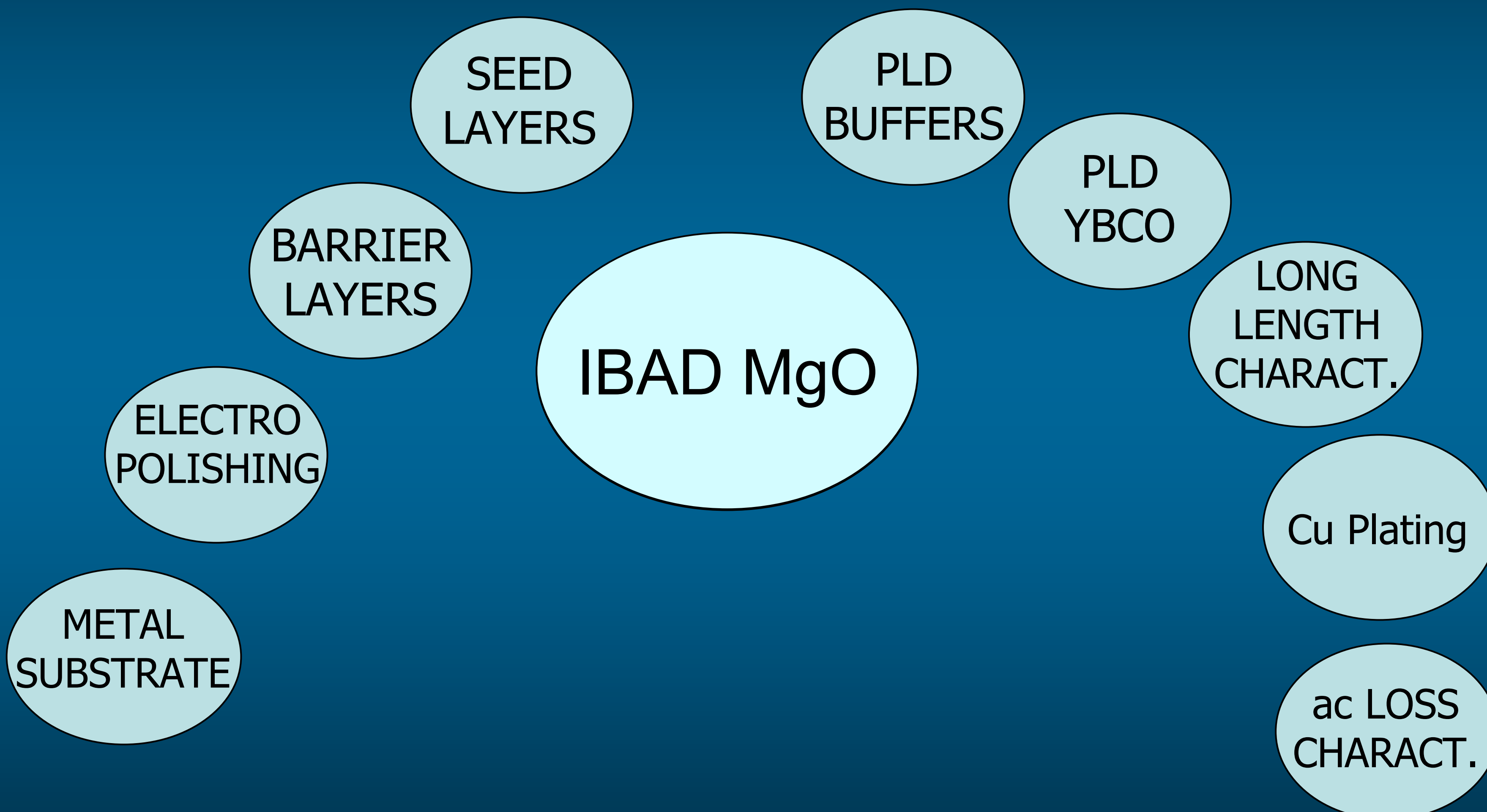
Outline

- Introduction
- Electropolishing
- Barrier layer
- Ion scattering setup
- IBAD-MgO
- Reactive evaporation
- PLD of buffers and YBCO
- New PLD heater
- Cu stabilization
- ac losses
- Summary slides

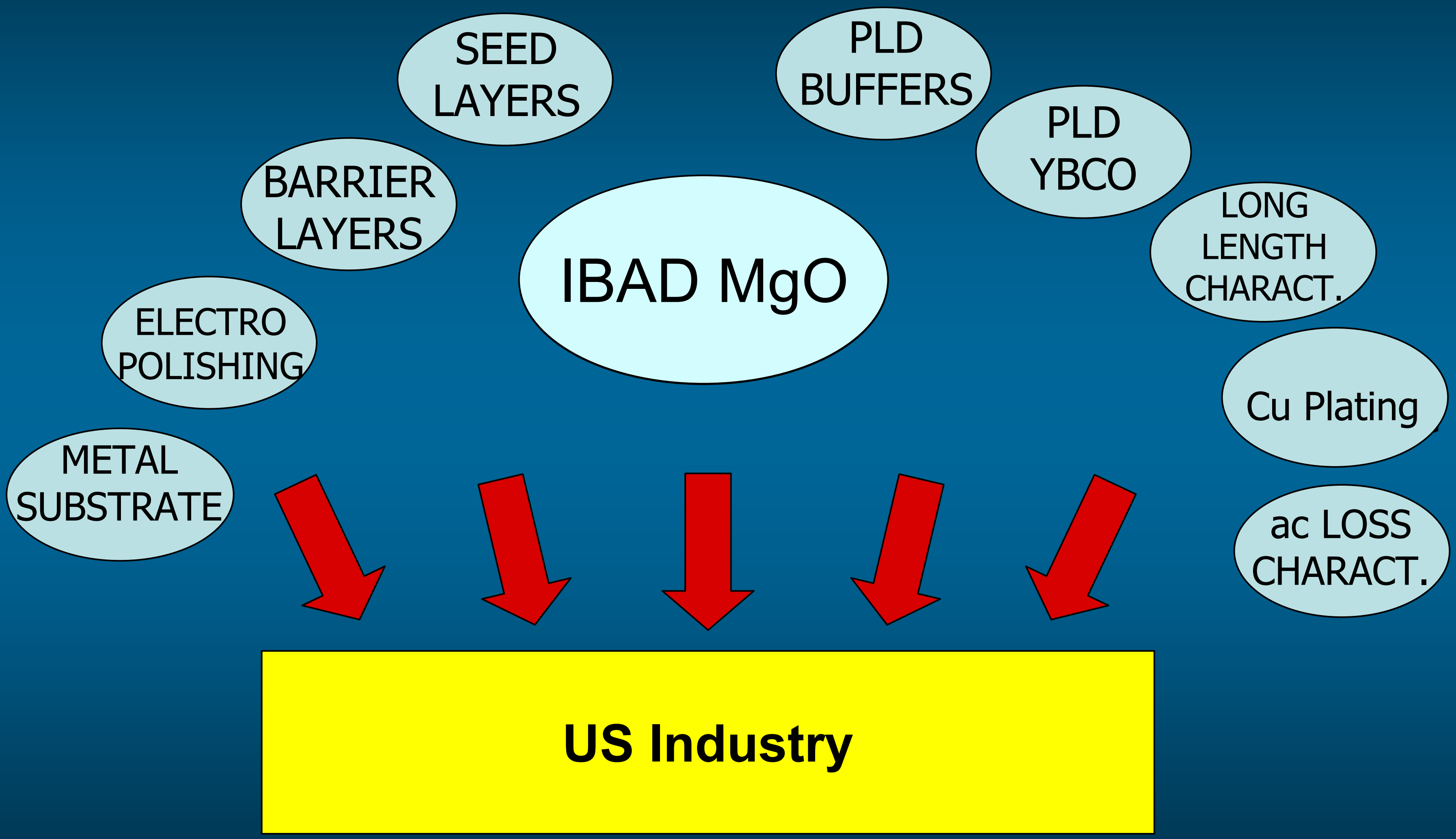
Introduction

- Los Alamos initiated the Coated Conductor (CC) research in the US 10 years ago
- LANL is accelerating development of CC and supporting the industrial effort by developing cost-effective processes and high-performance wire
- For this purpose an effort in continuous fabrication is required, coupled with state-of-the-art characterization facilities
- IBAD-MgO is the best texturing process; PLD-YBCO is our choice for demonstration of CC

LANL Develops Methods and Technologies for Fabricating and Characterizing CC's



LANL Transfers Methods and Technologies for Fabricating and Characterizing CC's



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LANL innovative Technologies Support Industrial Effort

LANL

- Electropolishing
- IBAD-MgO
- Materials char.

New technologies

- Alternate deposition methods
- Nano-processing
- Low ac-loss Coated Conductor
- 2000 A Coated Conductor

American Superconductor

- Ni passiv. layer
- Materials char.
- IBAD-MgO
- positional Ic
- Electropolishing

SuperPower Inc.

- IBAD-MgO
- Materials char.
- positional Ic
- IBAD-YSZ
- PLD Buffers/HTS
- Electropolishing

OXFORD INSTRUMENTS

- Materials char.

MetOx

- IBAD-MgO
- PLD buffers

Los Alamos CC Continuous Processing - FY04

- **Electropolishing**
  - polished > 2 km of tape; wider tapes
- **IBAD**
  - processed 400 meters of tape
  - added new *in situ* monitoring: **Ion scattering** and 2^{nd} RHEED
- **PLD**
  - processed 100 meters of tape
  - achieving 200 A results
- **Reactive coevaporation system operating**
- **Significant increase in collaborations**

Electropolishing System producing long lengths and on wider tapes

- Excellent polishing results on Hastelloy: 1 nm RMS roughness on 5 x 5 μm scale
- New system set up for examining different materials and electrolytes: more reliable solution
- Conditioning of electrolyte solved
- Polished 4-mil and 2-mil 1-cm wide tapes; also polished 3 cm-wide tapes; preparing to polish 6-cm wide tapes
- Speed (one cell): 12 m/hr

Electropolishing System is serving needs of industrial efforts

- Two years ago we demonstrated the utility of electropolishing
- Within a year we transferred the technology to SuperPower for their manufacturing facility and they pursued further improvements
- American Superconductor expressed an interest in electropolishing RABiTS precursor tape for cleaning purposes
- LANL successfully polished long lengths of RABiTS precursor NiW tape for AMSC this year

SuperPower Inc.

Routine EP speed
18m/h last review

42m/h this year

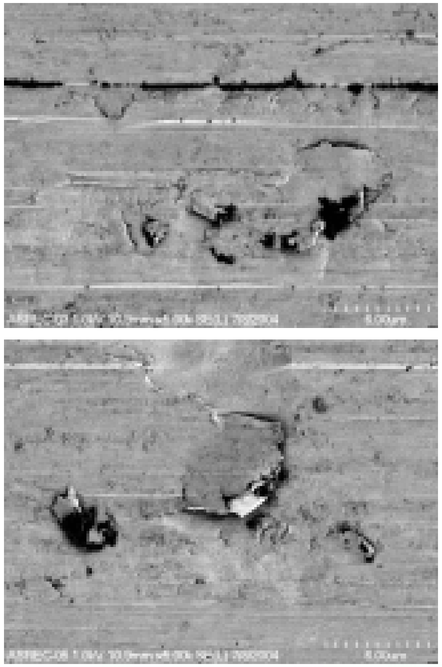
Ra (nm) < 1nm
5 µm AFM line scan by LANL

5x5 μm^2 AFM measured by SuperPower
no grain boundary with Nomarski DIC microscope

LANL Reel-to-Reel electro-polish used successfully for surface treatment of rolled Ni-5at%W strip

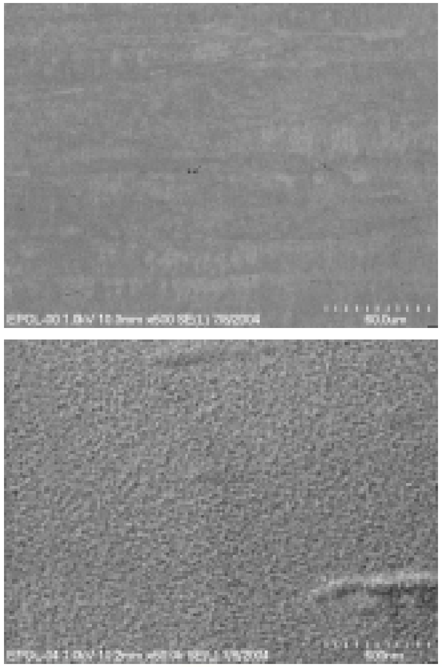
- NiW strip is purchased from vendor at 0.2 mm thickness, with:
  - surface contaminants (variable from run to run, metal and oxide particles)
  - relatively rough surface with folded-in material
- **Surface cleaning before further rolling is essential**

122 nm Ra



before e-polish

90 nm Ra

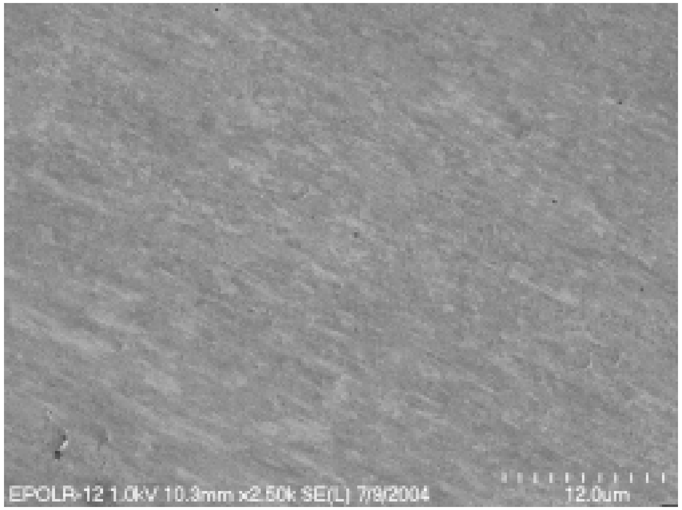


after e-polish

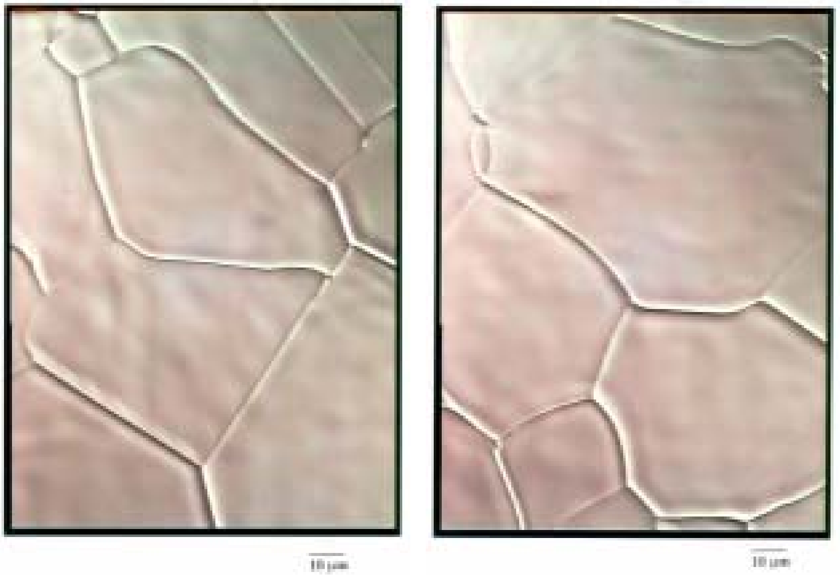
As rolled, and after texture anneal: clean surface

- Surface after rolling to 0.06 mm is clean and smooth (12 nm Ra)
- Surface after texture annealing
  - No contaminant with LM or AFM
  - Surface roughness inside grains <0.2 nm Ra

e-polish at 0.2mm + roll to 0.06mm clean and smooth: 12 nm Ra

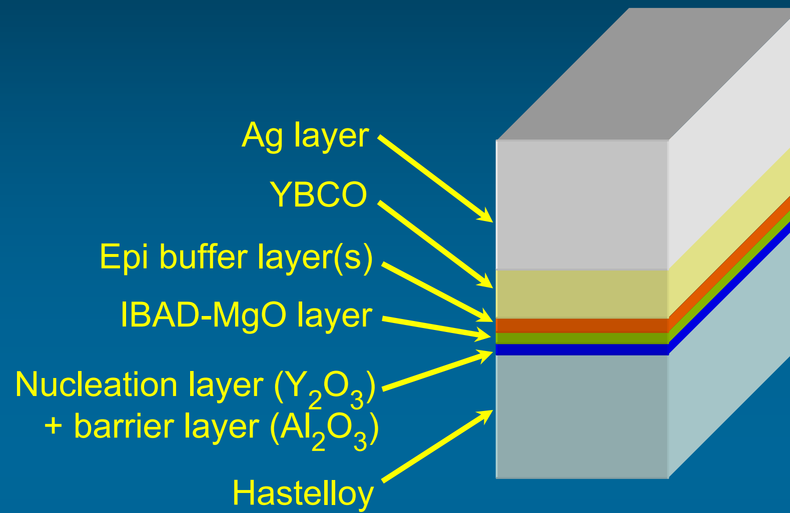


e-polish + roll + texture anneal: clean - <0.2 nm Ra inside grains



LANL Coated Conductor

- IBAD-textured MgO template on a Ni-superalloy
  - IBAD layers deposited by e-beam evaporation at RP
- Pulsed-laser deposited buffers and superconductor



Los Alamos Scale Up Effort:

Tape Prep → Nucleat. layers → IBAD texturing → EPI layers → HTS layer → Normal metal

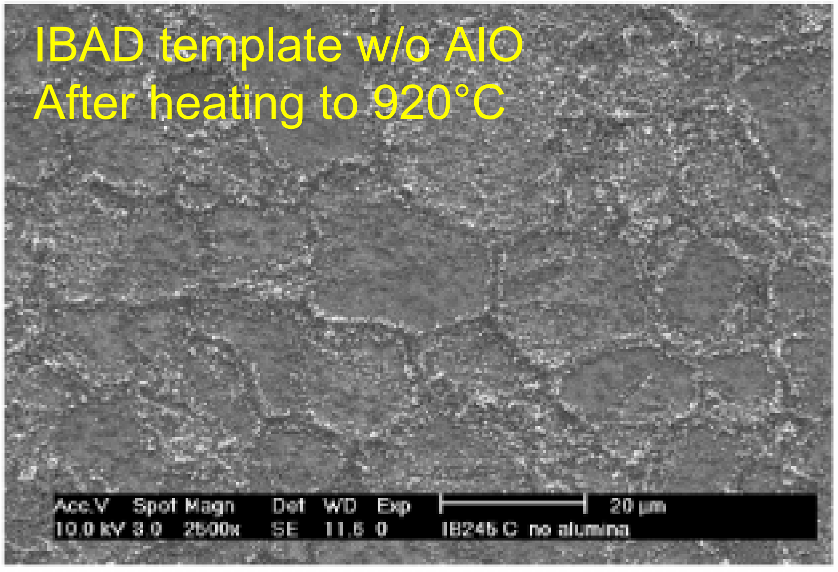
- Electropolishing System: Tape Prep
- IBAD/e-beam System: Nucleat. layers, IBAD texturing, EPI layers
- PLD System: EPI layers, HTS layer, Normal metal

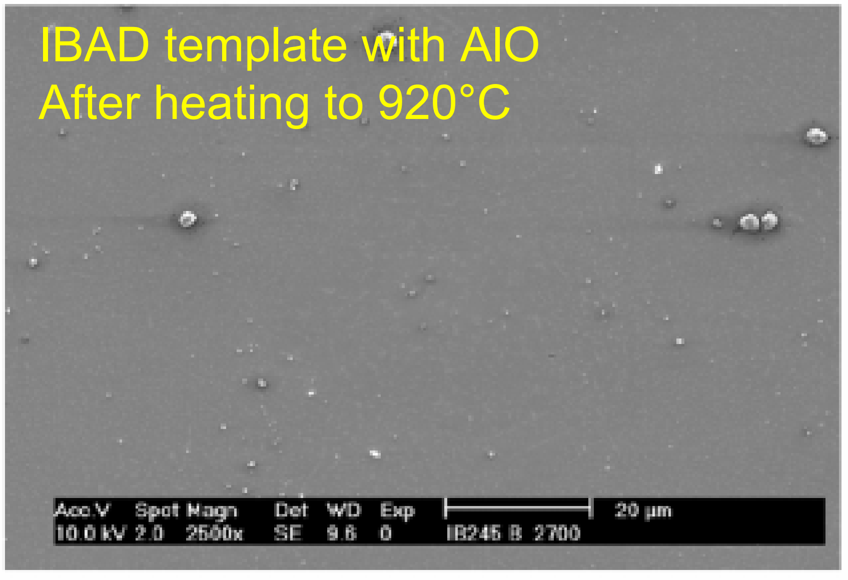
Layers are needed for different functionality

- Barrier layer prevents interdiffusion
  - Utilize a-Al_2O_3 with ion beam assist for densifying
- Nucleation layer needed for IBAD-MgO
  - Y_2O_3 layer unique to LANL process
  - very thin layer (~ 5 nm)
- IBAD-MgO provides in-plane texture
- Homoepitaxial MgO layer relieves the strain in MgO
- Epitaxial buffer layer provides good lattice match to YBCO
  - Perovskite materials work best for buffer layers
  - Improves texture significantly

Good barrier layer prevents diffusion of metal species from substrate

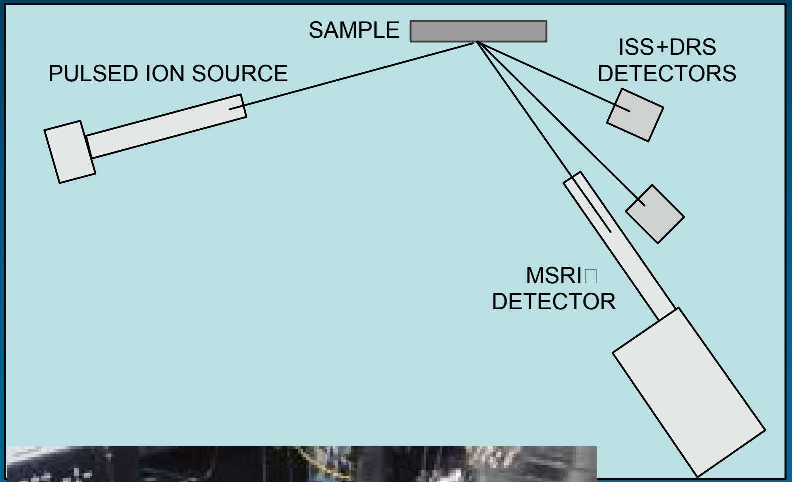
- LANL program developed and successfully used a thin a-Al_2O_3 layer (80 nm) as a barrier layer



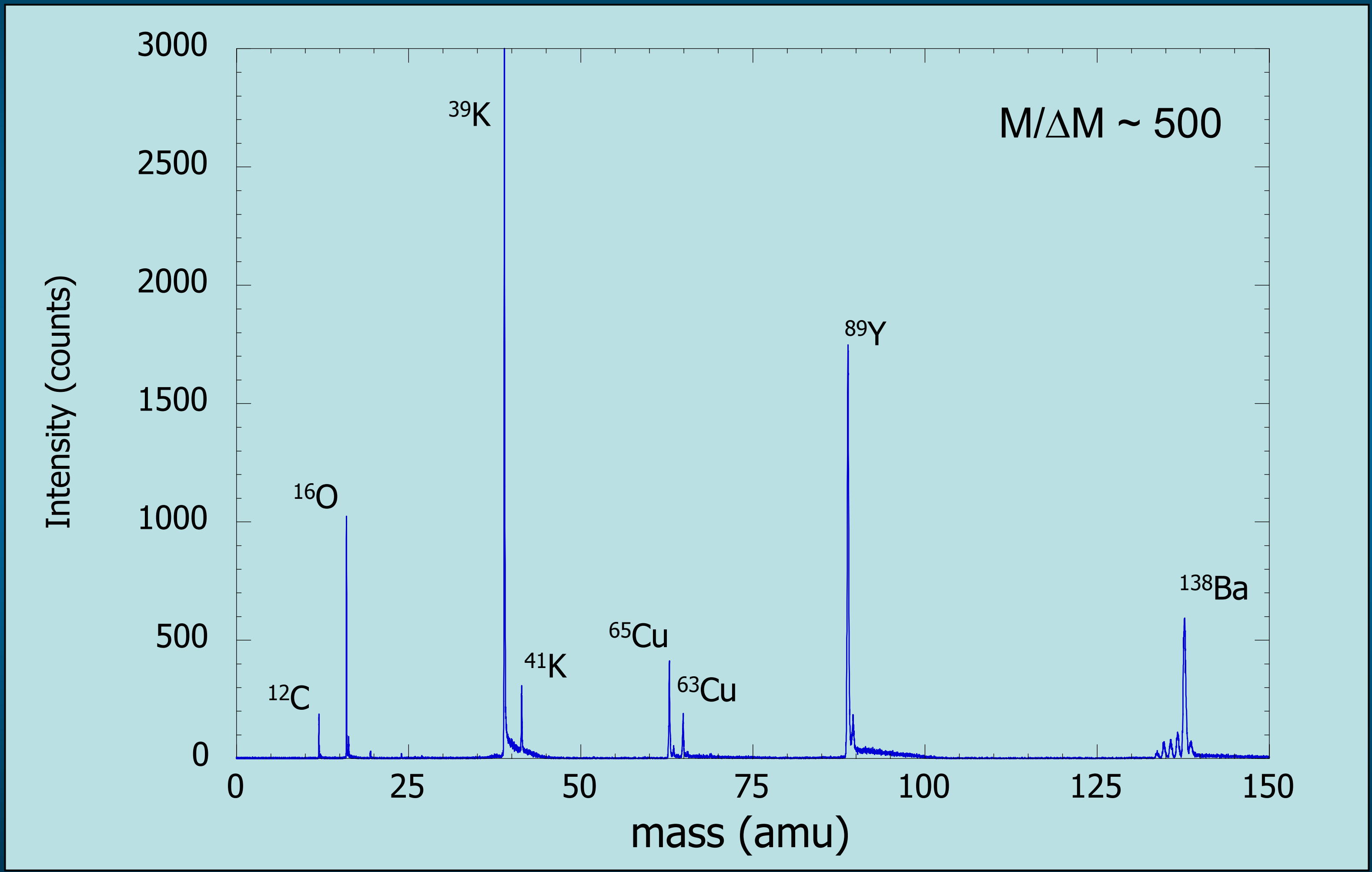


Ion Scattering Spectrometry added to the IBAD system for in situ diagnostic

- Time-of-flight ion scattering spectroscopy (ISS), recoil spectroscopy (DRS), and reflectron mass spectrometer (MSRI)
- MSRI similar to SIMS but capable of high-resolution surface analysis at higher ambient pressures
- Allows for real-time, *in-situ* analysis of the film growth process
- Non-destructive

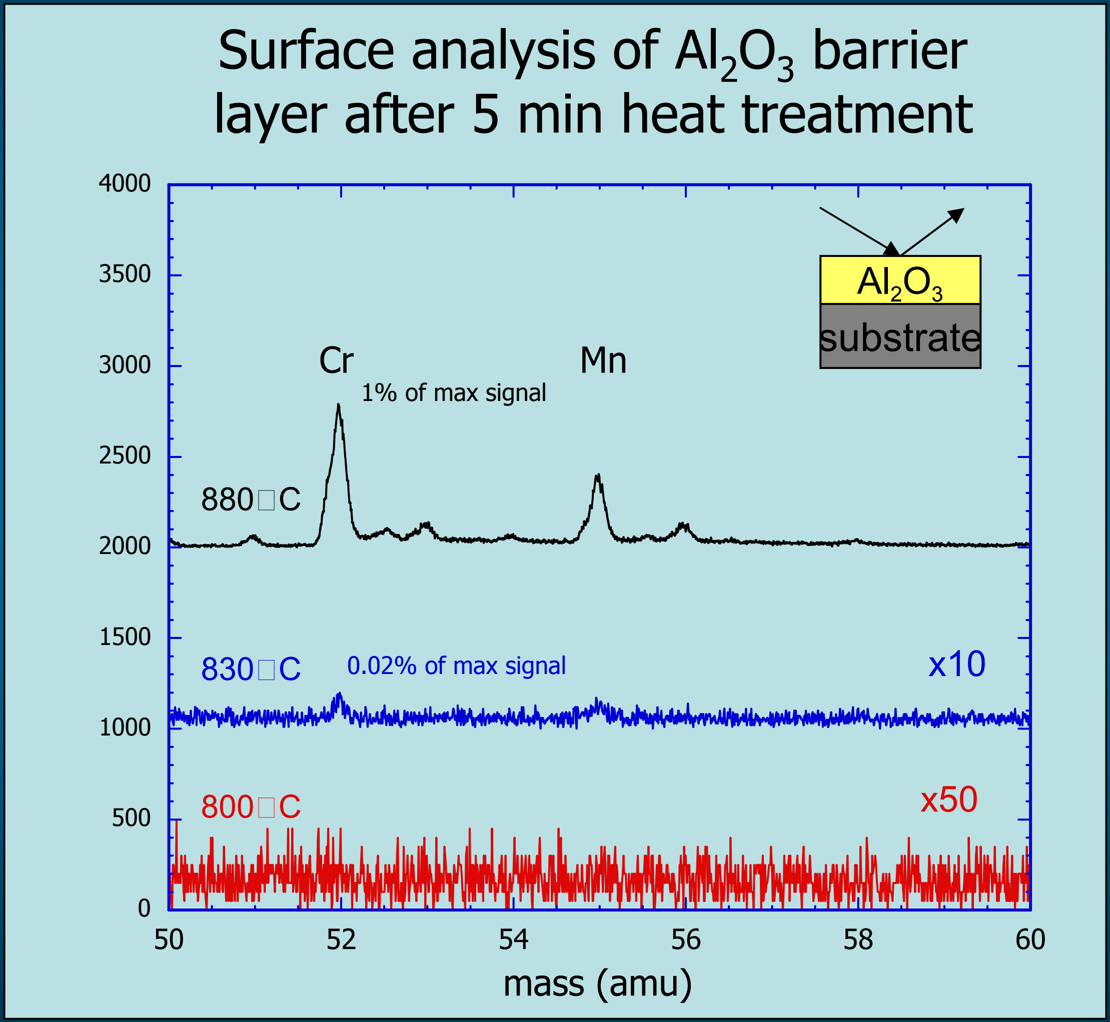


TOF-ISARS - Mass spectroscopy of YBCO



Ion Scattering shows diffusion of metal species through the IBAD layer stack

- ISS used to analyze metallic species diffusing from the Hastelloy substrate and through the barrier layer
- Tests done upon heating in oxygen
- Fully oxygenated alumina improves as barrier



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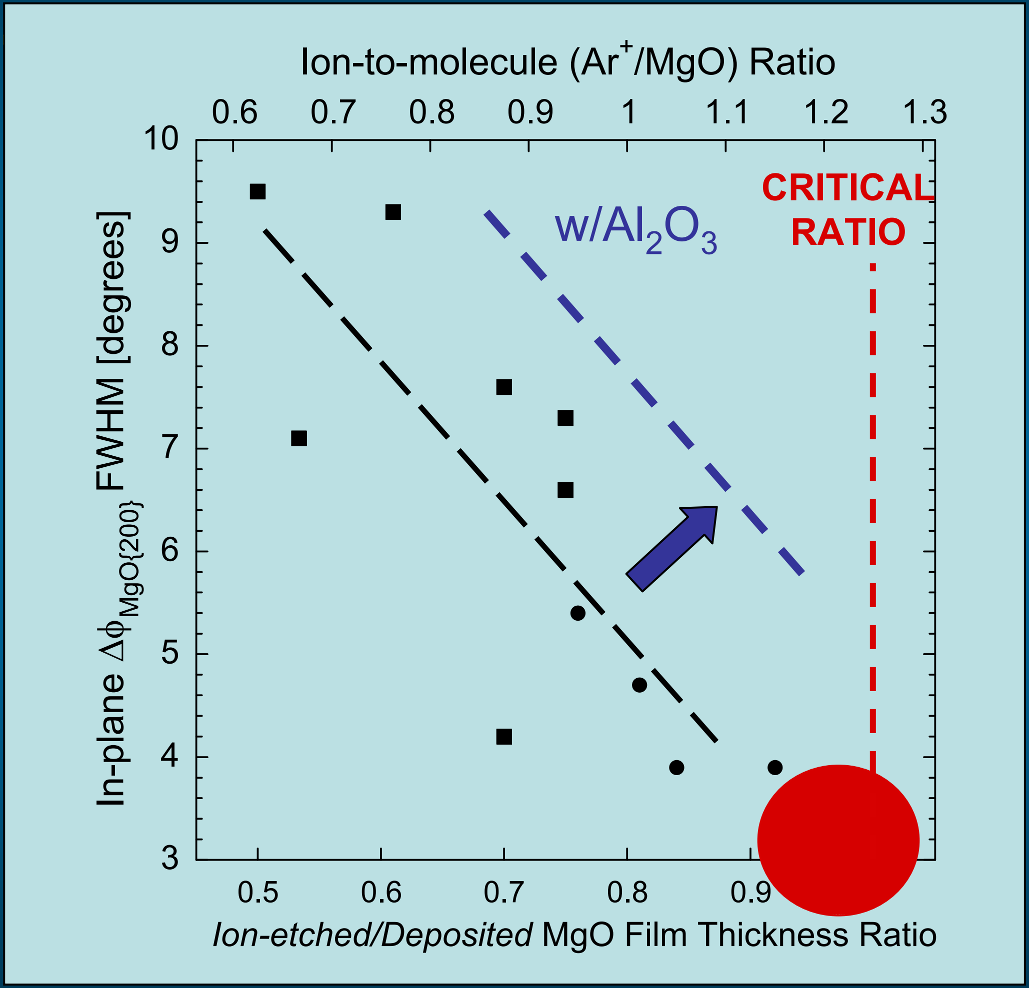
IBAD-MgO Status

- IBAD-MgO by e-beam deposition is very stable
- Best samples made exhibit FWHM in-plane < 4°
- Long lengths fabricated with in-plane texture of 6–8°
- Multiple 10-15 meter pieces made; 4-mil and 2-mil

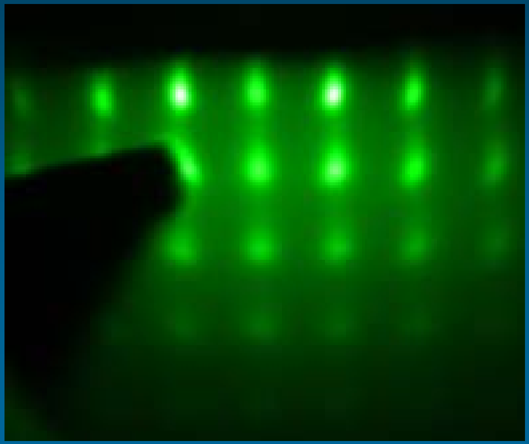
Last year we showed the importance of the ion-to-molecule ratio during IBAD texturing

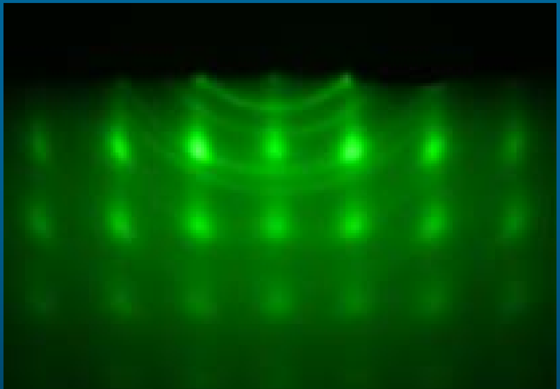
- In-plane and out-of-plane texture improves as the ion-to-molecule ratio, *r* increases
- Accessible range of ratios depends on the nucleation surface
- On alumina texture was typically not as good (1–2° worse in-plane)



Alumina barrier layer issues

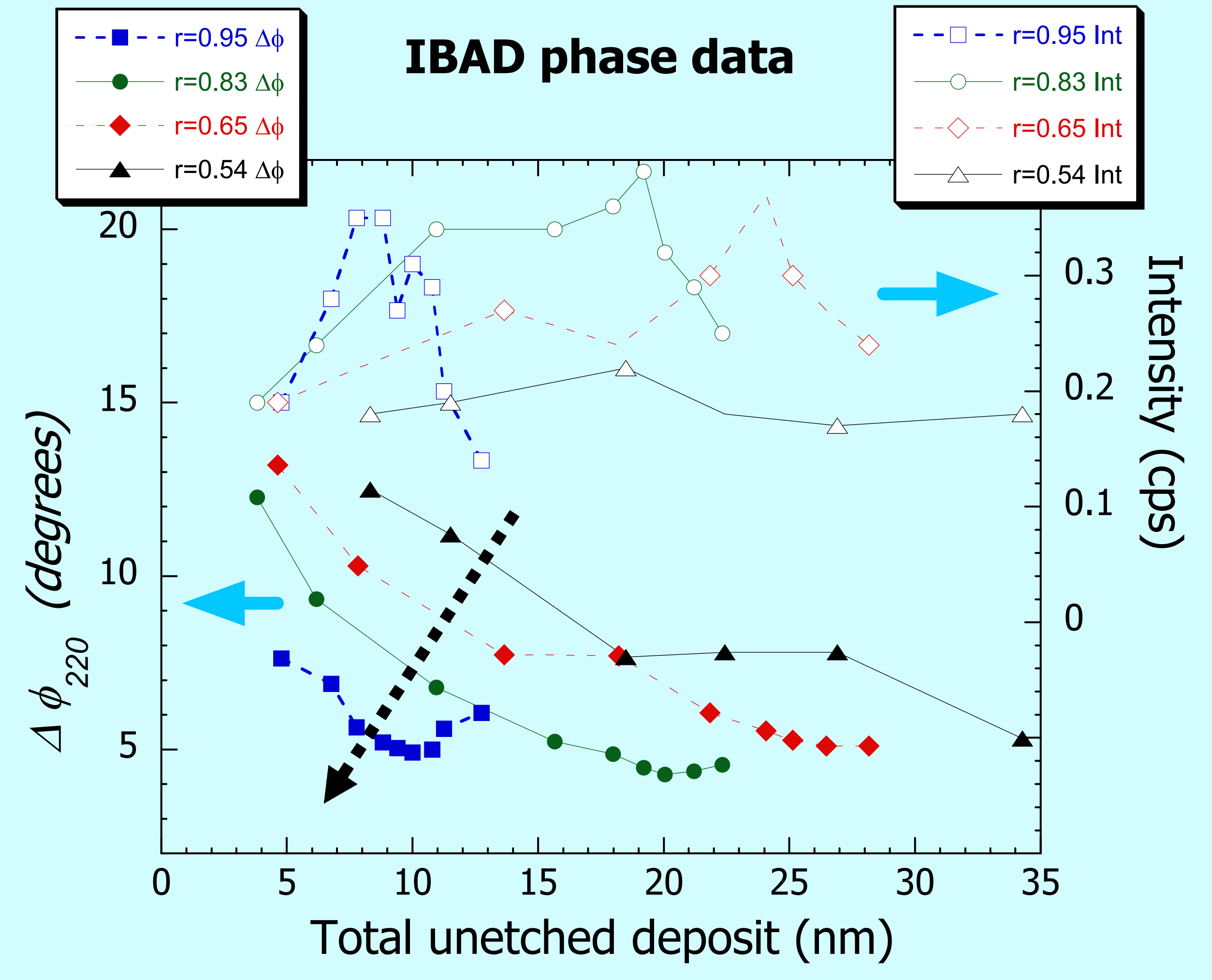
- IBAD a-Al_2O_3 worked well for us as a barrier layer at thicknesses of 60 – 80 nm; demonstrated I_c in YBCO up to 200 A
- We made about 400 meters of IBAD (over 100 runs) in the last year
- Results were not always repeatable and as good as we would like
  - We get polycrystalline rings in the RHEED and reduced intensity in the XRD, depending on the condition of the alumina deposit
- Alumina has 2 drawbacks: slightly poorer texture and occasionally worse crystalline fraction in IBAD-MgO





IBAD-MgO with rings

IBAD-MgO texture depends on ratio *and* total deposit thickness

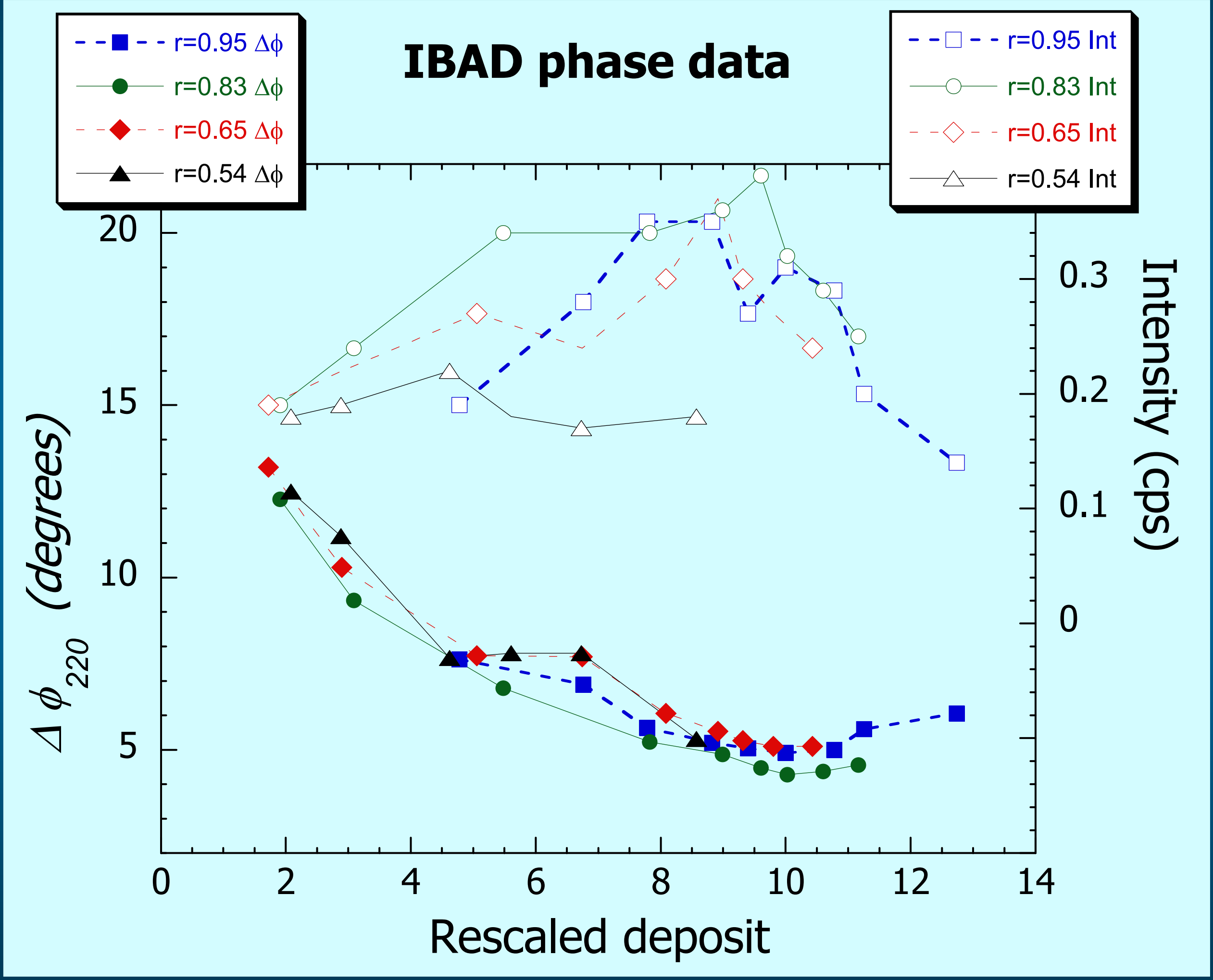


Details depend on growth surface!

Process is more robust at higher ratios

Texture curves exhibit scaling

IBAD-MgO texture scales with ratio and total thickness



These curves are for flat deposition profiles

We believe that it can be improved by optimizing the profile

More research needed

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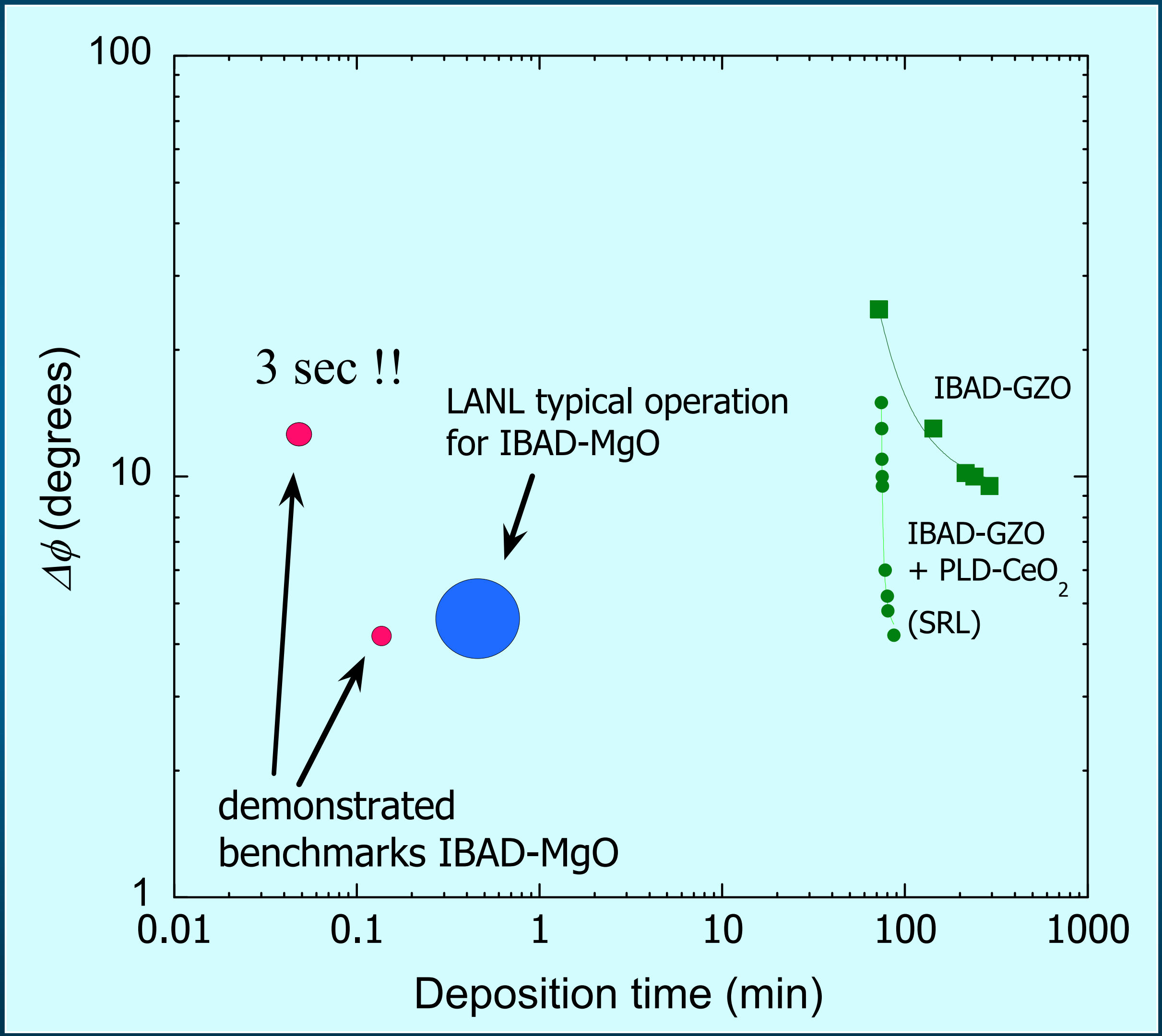
IBAD-MgO demonstrated at a speed of 3 cm/sec or 100 m/hr



Speed of IBAD-MgO Process

- We have demonstrated 100 meters/hour with e-beam evaporation
  - Speed limited by the ion gun utilized
- We can extrapolate (!) to 5x wider tape yielding 500 m/hour of cm-equivalent tape (with same ion gun)
- Three ion guns could be placed in series to increase the throughput to **1.5 km/hour** of cm-equivalent tape

Need high-throughput manufacturing processes for CC: 1 km/hr

	Electro-polishing	Barrier layer (AlO)	Nucl. Layer (YO)	IBAD -MgO	Epi-MgO	Buffer layer (LMO)	YBCO (PVD)	Normal metal (Ag/Cu)
Demonstrated speed cm-m/hr	36	4	15	100	10	20	15	-
Capable speed cm-m/hr	150	20	150	500	100	?	?	*5*
Status	✓	?	✓	✓	✓	?	?	✓

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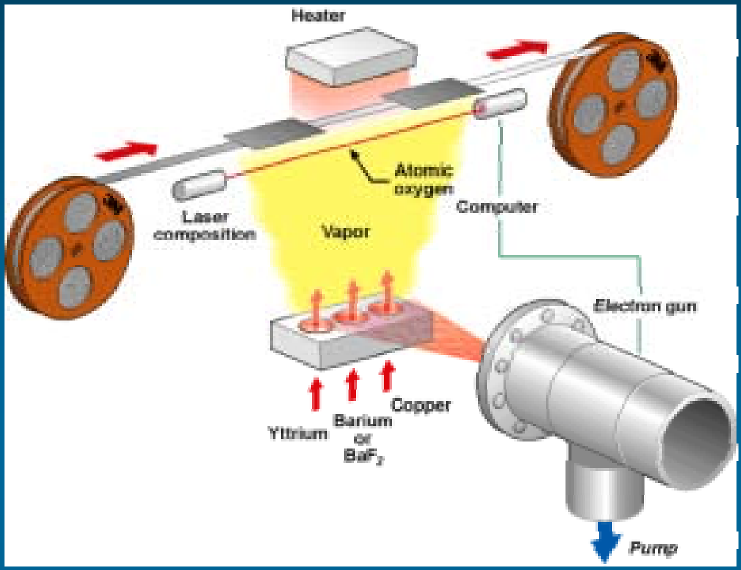
Reactive Coevaporation is being tried for fast deposition of superconductor

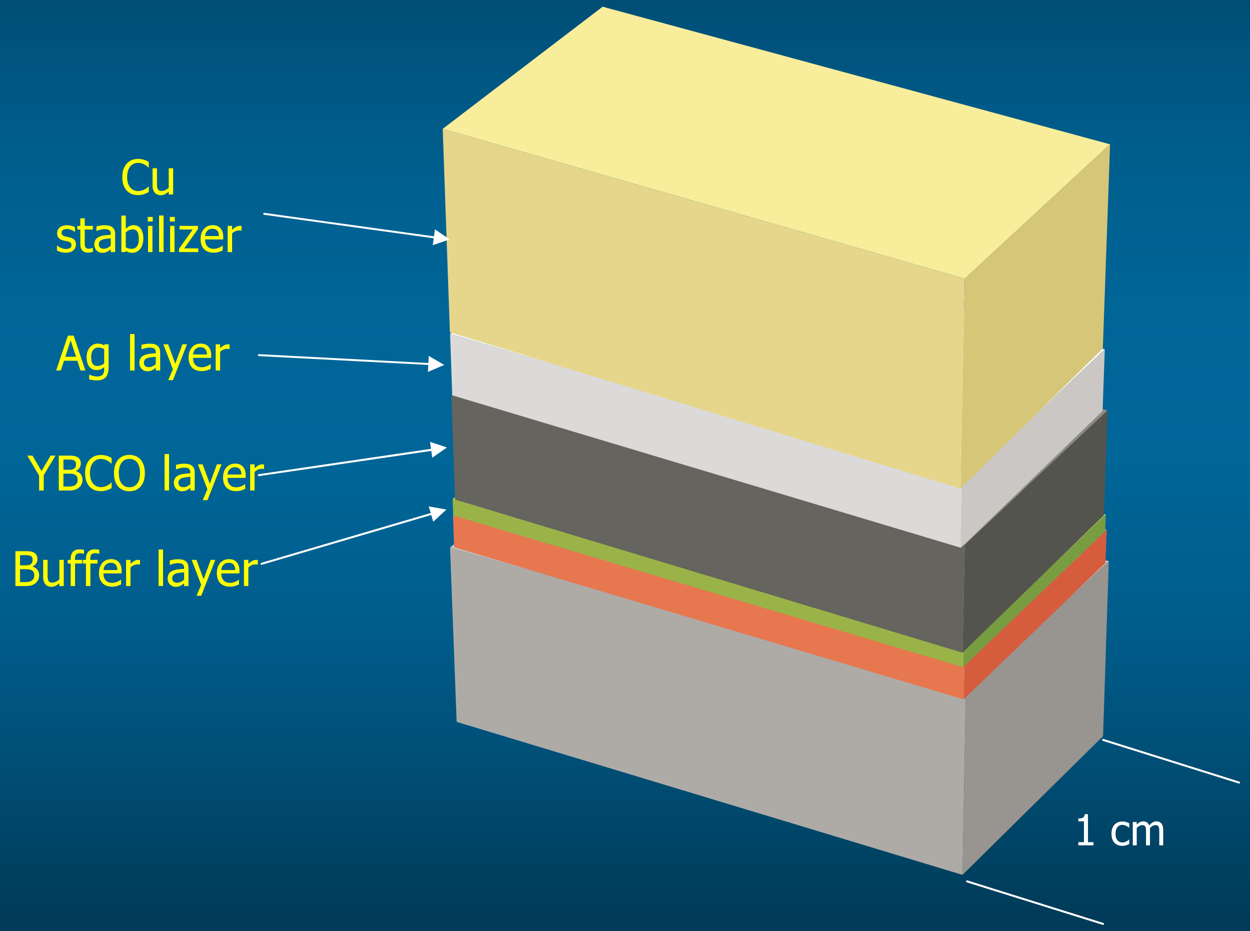
- Need a high-throughput process for *thick* YBCO film deposition to complete CC wire manufacturing
- Evaporation is known to be able to deposit at very high rates
- Reactive coevaporation (RCE) is a proven uniform process for deposition of YBCO on wafers in electronics
- Collaboration with Stanford University is testing out feasibility of high-rate YBCO deposition by RCE for wire manufacturing

Co-evaporation system is installed and operating at the Los Alamos Research Park



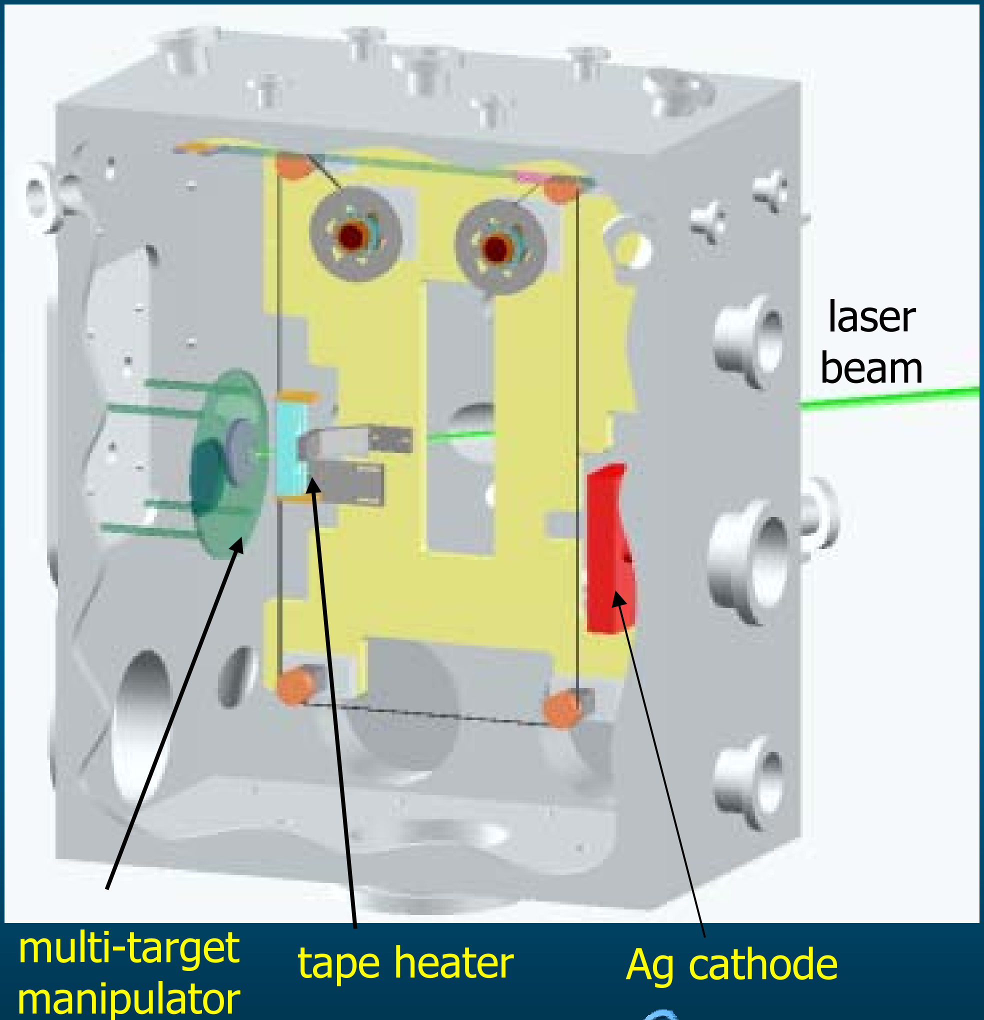
- e-beam evaporator with computer controlled scanning
- Laser-atomic absorption spectroscopy for accurate rate control
- Uses inexpensive source materials

LANL's Coated Conductor Architecture



LANL's reel-to-reel PLD system

- 200 W XeCl (308 nm) excimer laser
- Four 4" targets for deposition of a variety of oxide layers
- Quartz lamp heater
- *In situ* adjustment of tape position with respect to laser plume
- Silver deposition integrated



Significant progress has been made over the past year - at the 2003 Annual Review:

- In 2003 our results on 2 μm YBCO/50 nm LMO/IBAD template/Hastelloy
  - 178 A across 1 cm
  - 120 A across 10 - 20 cm
  - 50 A across 1.1 m
  - Microbridges – 1.1 MA/cm^2

- Encouraging results, yet far from potential

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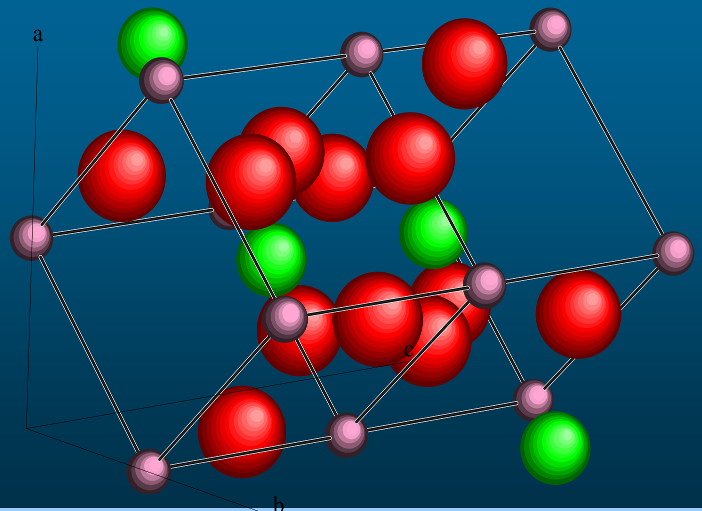
What is important for the PLD-deposited layers?

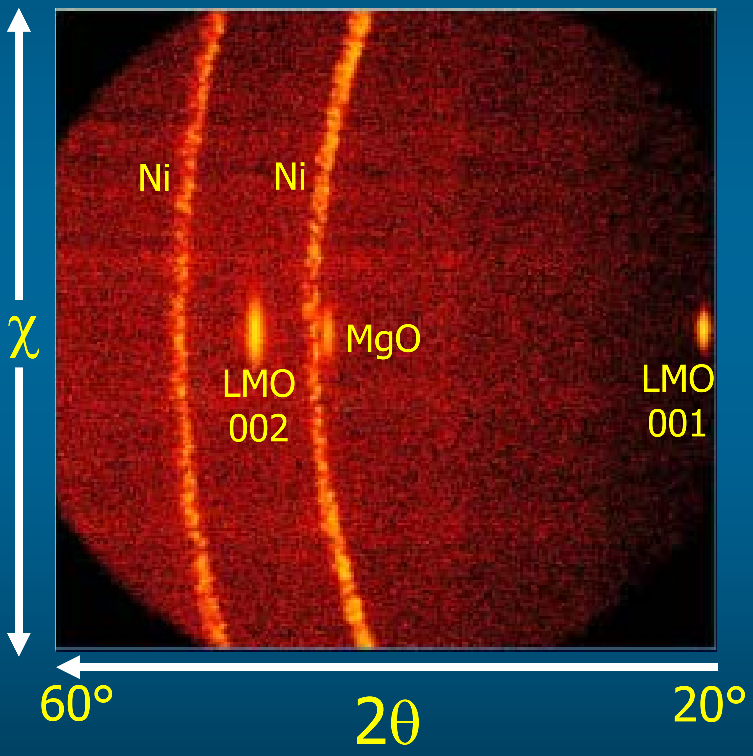
- Uniform, dense microstructure
- Reproducibility from run to run
- Uniformity over long lengths
- Fast
- High I_c, J_c!

Currently we are using $LaMnO_3$ and $SrTiO_3$ for our PLD-deposited buffer layer

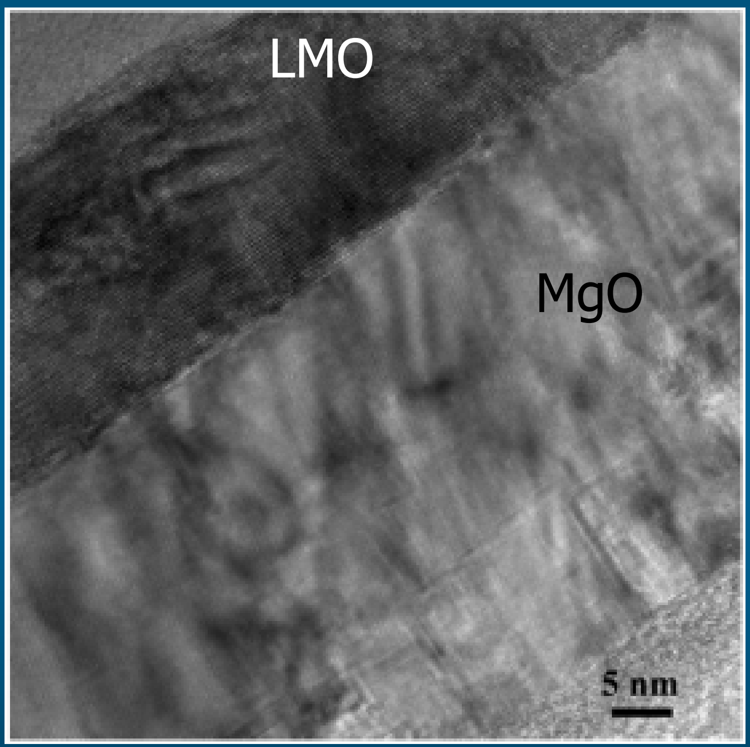
- $LaMnO_3$ buffer layer
  - Pseudo-cubic perovskite (110 spacing = 0.3985 nm)
  - Low deposition temperature
  - Wide temperature window
  - High deposition rate (0.1 nm/shot)
  - Up to 21 m/hr

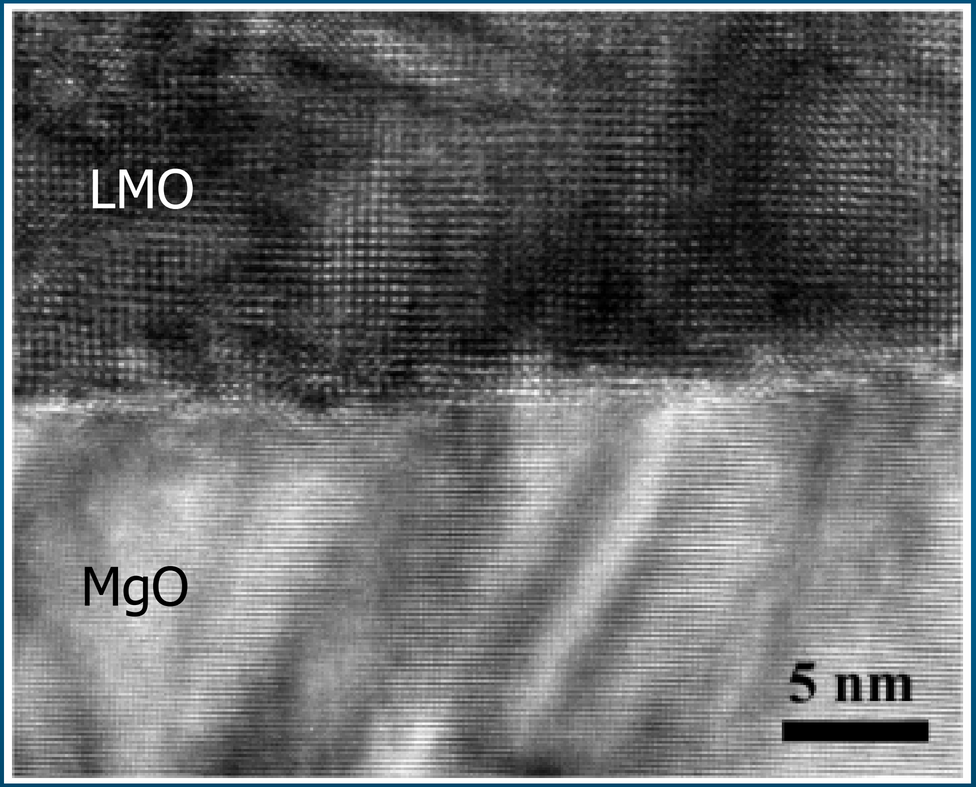


Bruker GADDS system θ-2θ frame

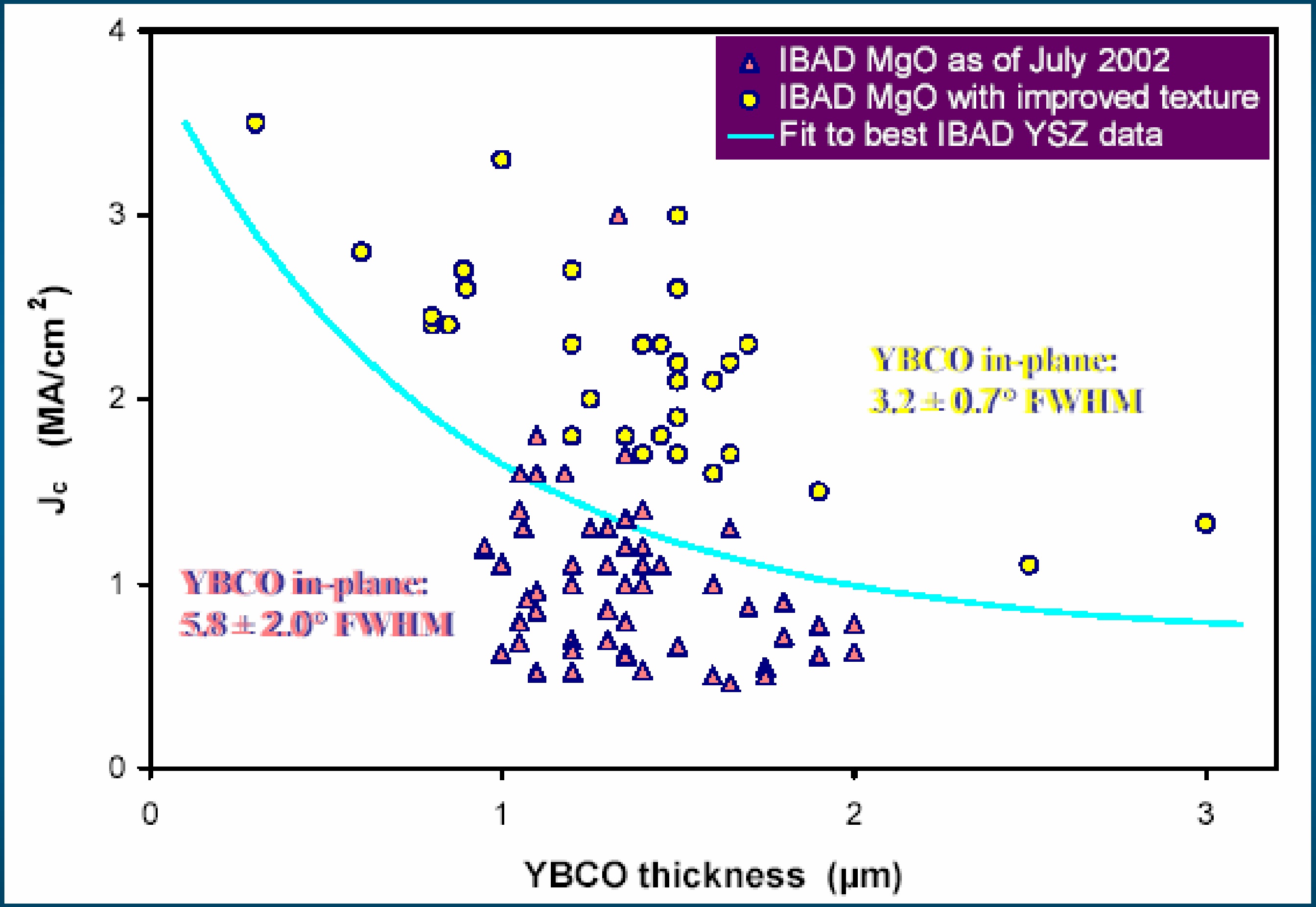


TEM data on the LMO layer indicates a clean interface with the underlying epi-MgO template





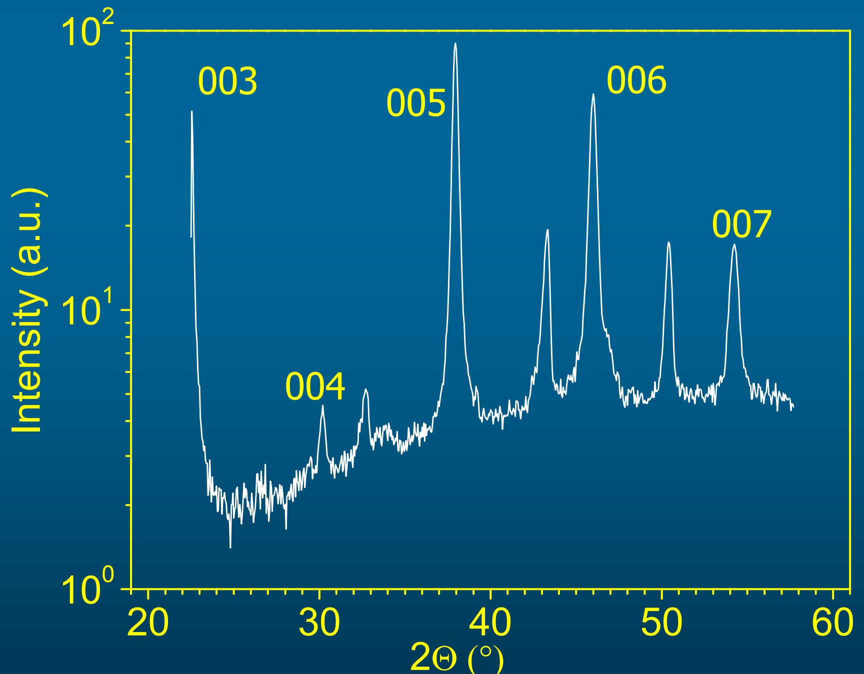
Improved texture results in consistently higher performing samples on IBAD-MgO – this is what is possible



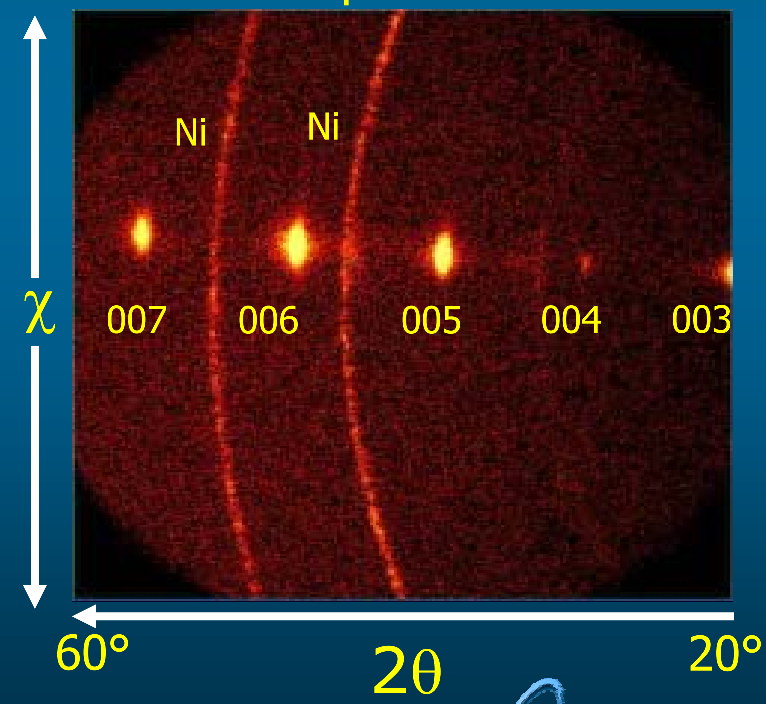
Acknowledgements to Steve Foltyn and Paul Arendt

Structural characterization indicates good *c*-axis texture throughout the stack

- Typical rates/conditions
  - LMO @ 13.5 m/hr, 50 nm thick, 200 mTorr O_2, 15 Hz
  - YBCO @ 1.8 – 2.7 m/hr, 1-2 µm thick, 180-200 mTorr O_2, 80-200 Hz

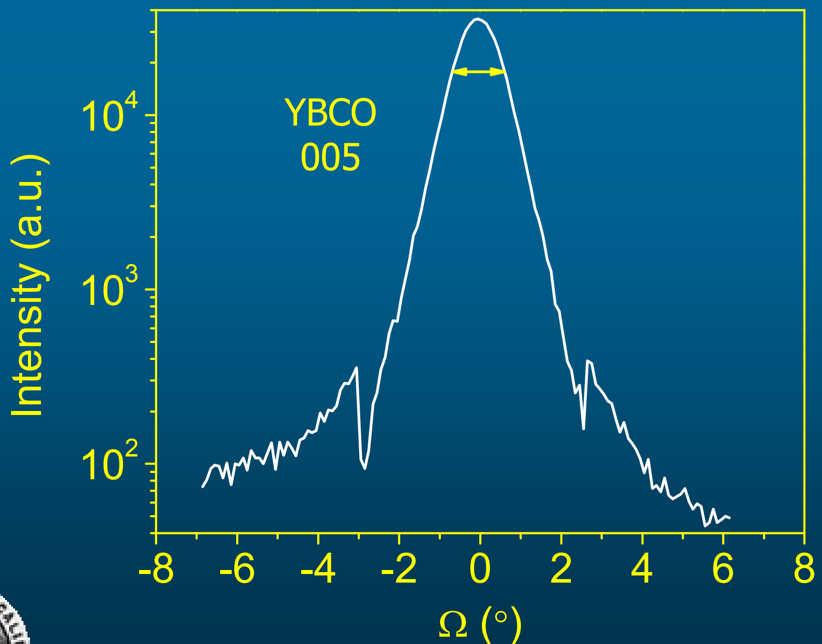


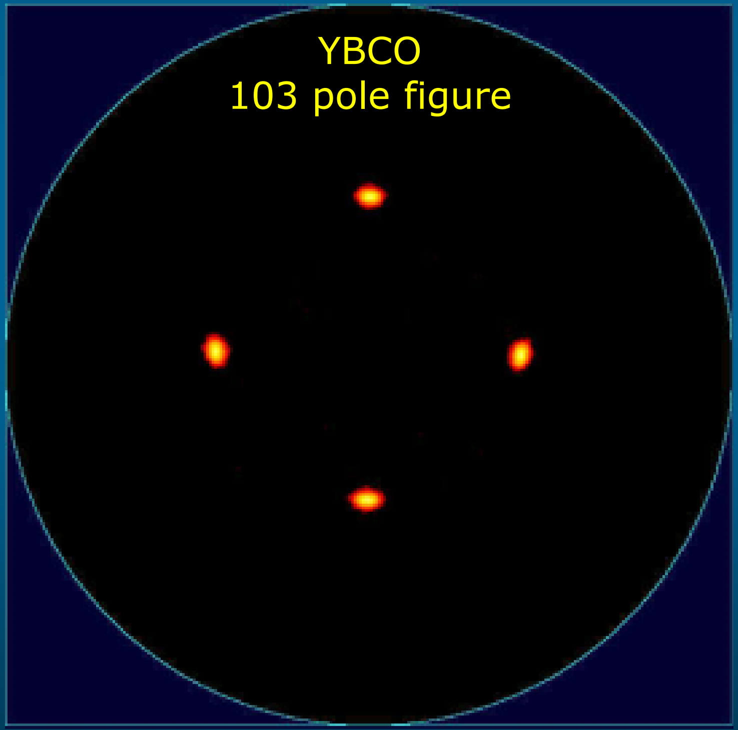
1.3 µm YBCO



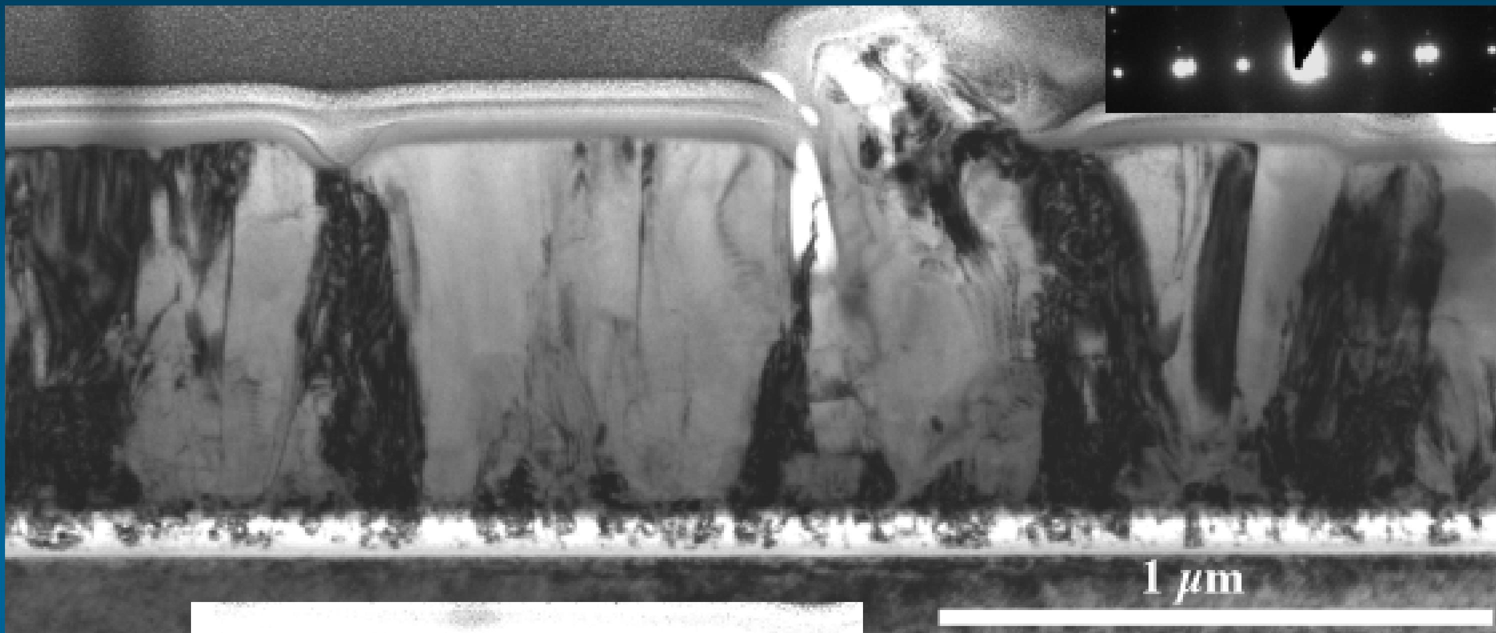
Texture measurements indicate comparable values for continuous deposition as compared to paste-up

- Continuously processed coated conductors show excellent texture/alignment
  - 50 nm LMO ϕ-FWHM ~ 5 – 6°
  - 1.5 μm YBCO ϕ-FWHM ~ 2.5 – 3.5°
  - 1.5 μm YBCO Ω-FWHM ~ 1.1 – 1.4°





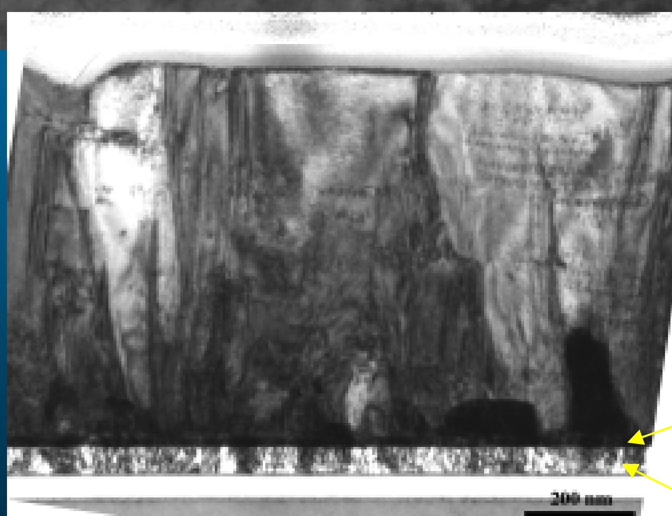
TEM data show the typical columnar microstructure for PLD-deposited YBCO films



YBCO

LMO

MgO



YBCO

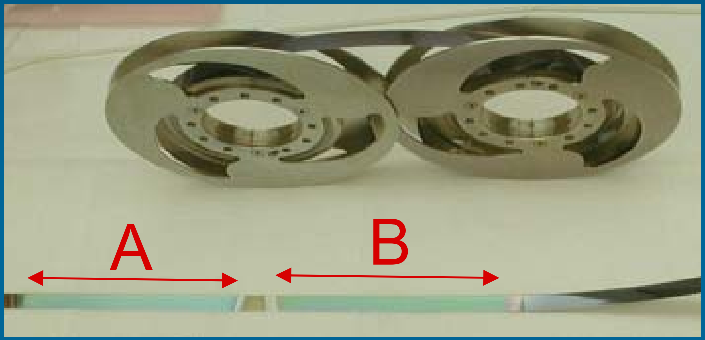
LMO

MgO

- $J_c \approx 1.9\ MA/cm^2$

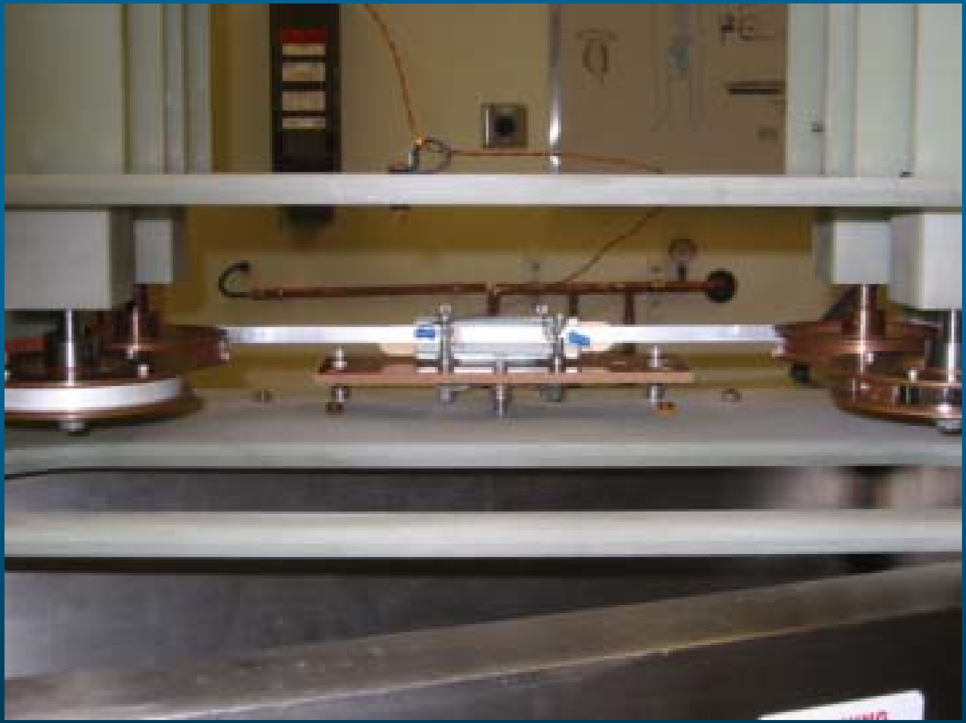
Continuous tape processing is ideal for exploring process parameters in a sequential way

- We can track positions from one process step to the next and develop a matrix of experiments for optimization
- For this to work, we need to measure I_c continuously



A and B have different buffer layers on top of IBAD-MgO

- Measurement length is 5 cm
- Step size = 2 cm
- Current fed in through outer reels



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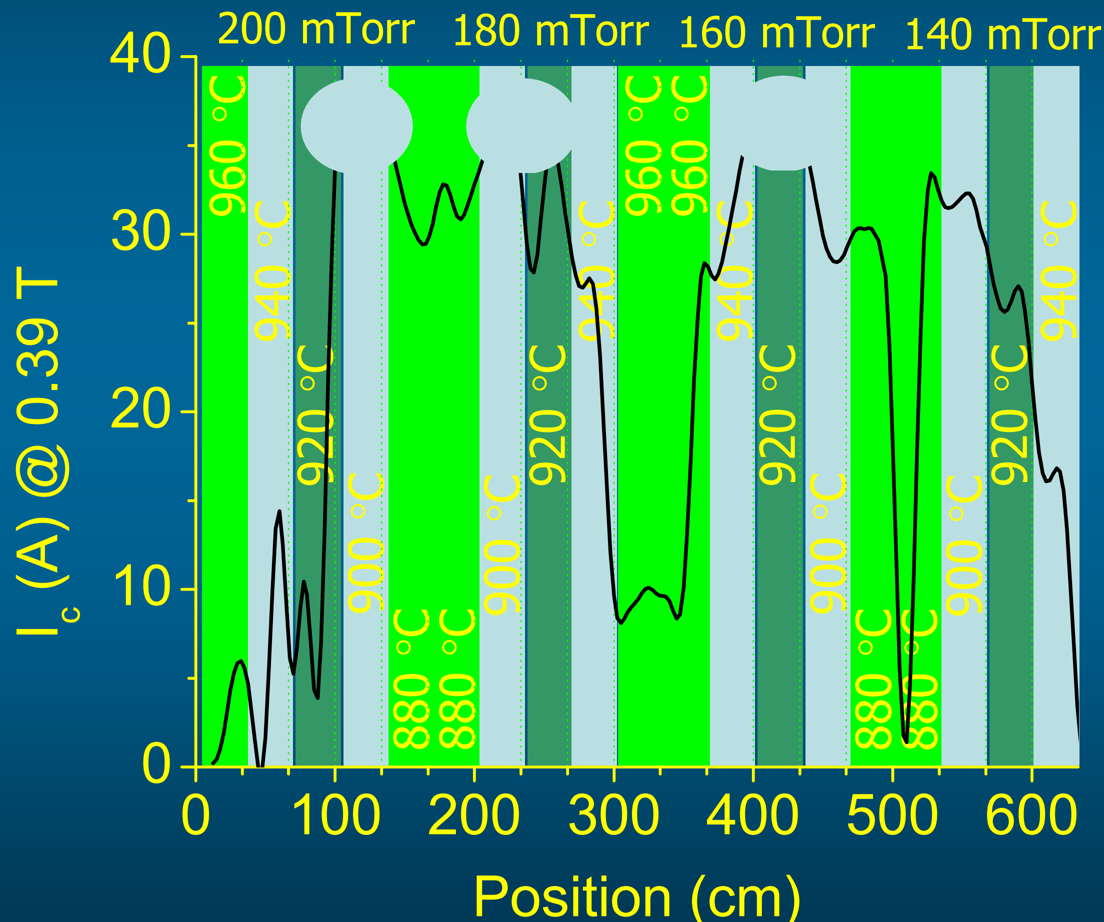
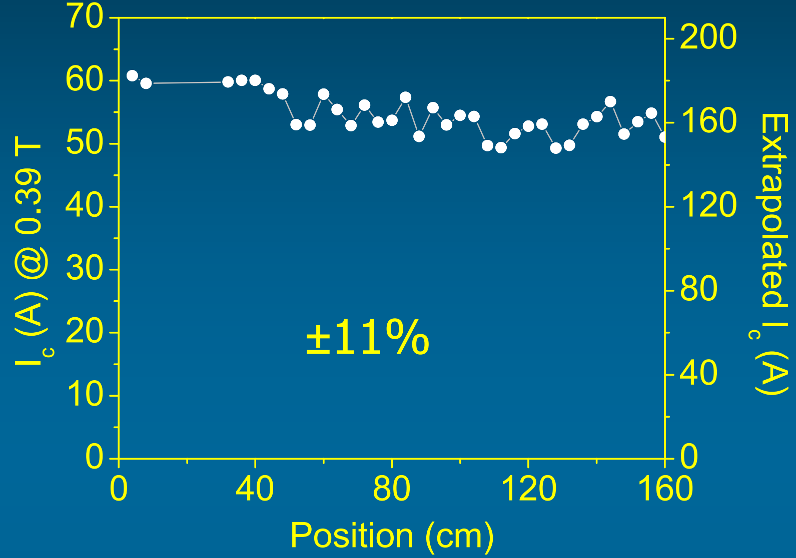
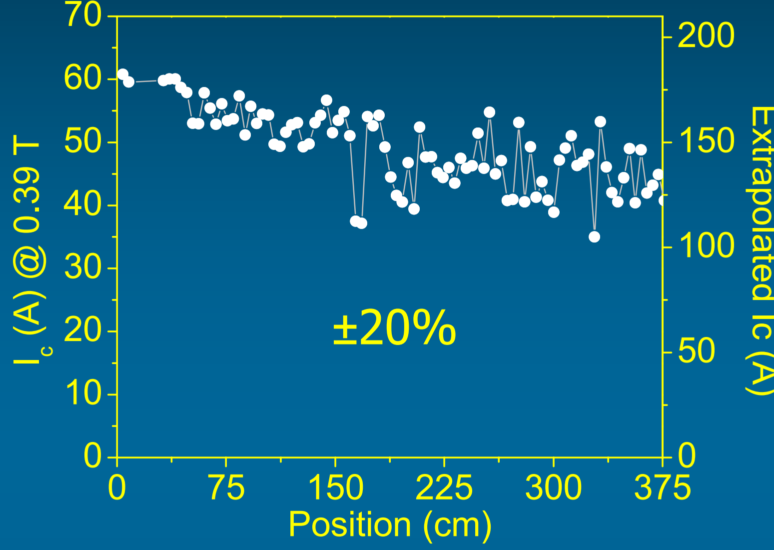
With this method, it's possible to isolate conditions which result in highest quality YBCO films

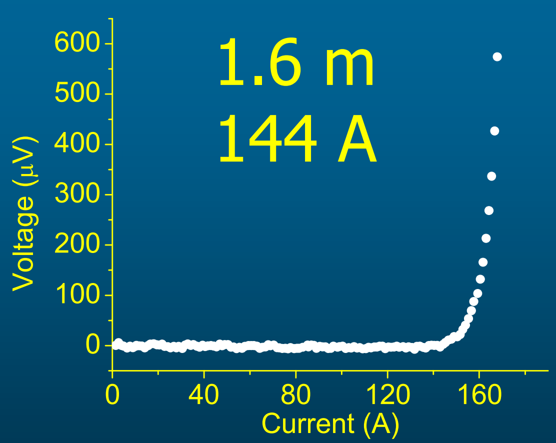
- Combinatorially process 6 m as a function of temperature and oxygen pressure
  - 900 – 920 °C (heater temperature, not substrate temperature)
  - 180 - 200 mTorr

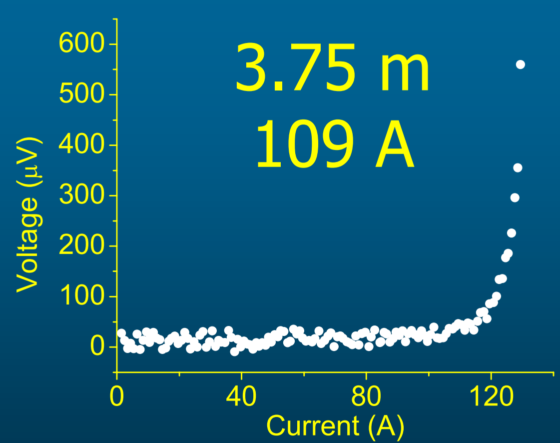


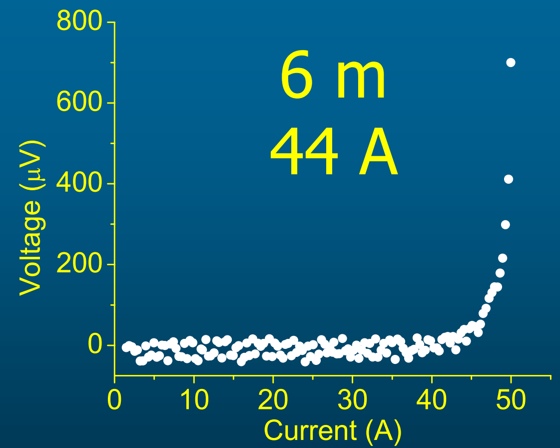
6 m IBAD-MgO-based Coated Conductor







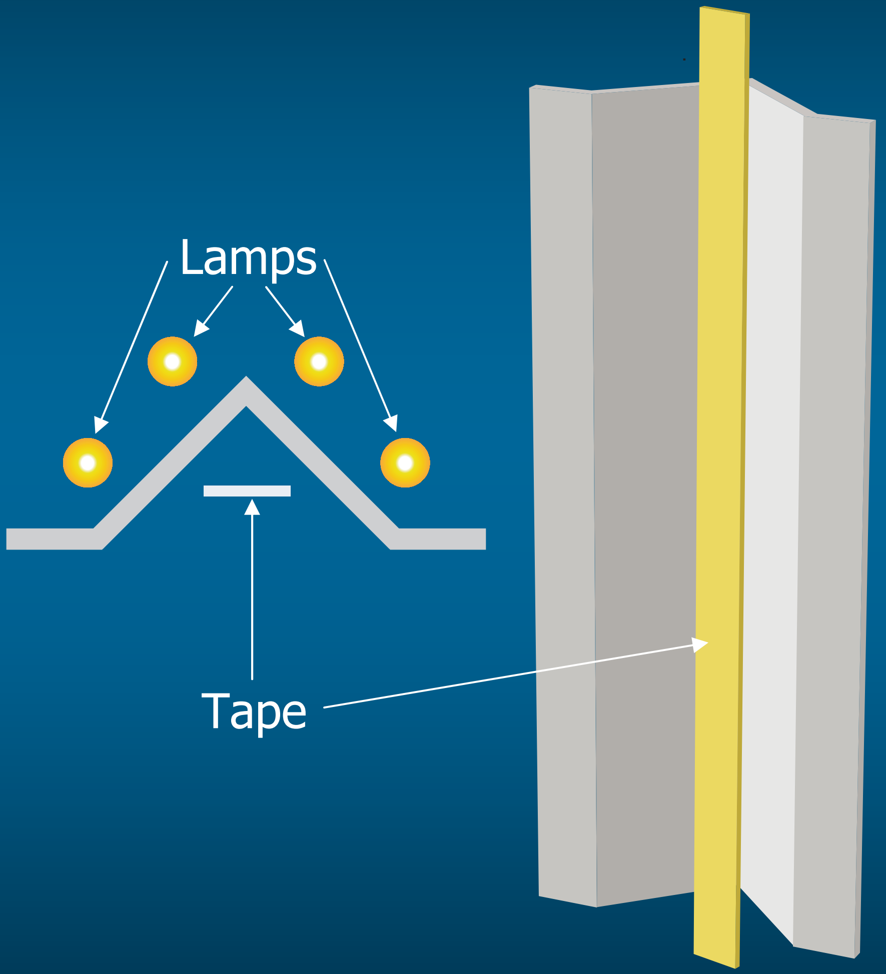




Measurements @ 75 K

Although the results were promising, the significant non-uniformity was not acceptable: reasons?

- Plume instability
  - Associated with unstable target raster mechanism
  - Use of a rotary magnetic feedthrough resulted in positional instabilities
  - Solved with a ferrofluidic feedthrough to a chain drive for the raster
- Heating
  - Old heater used 4 lamps axial to the tape heating a Haynes® 214 v-shaped susceptor plate



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New heater

- 9 separately controlled zones
- Quartz or Haynes® 214 susceptor plate
- Lamps are perpendicular to tape
- Designed for more uniform heating across the tape

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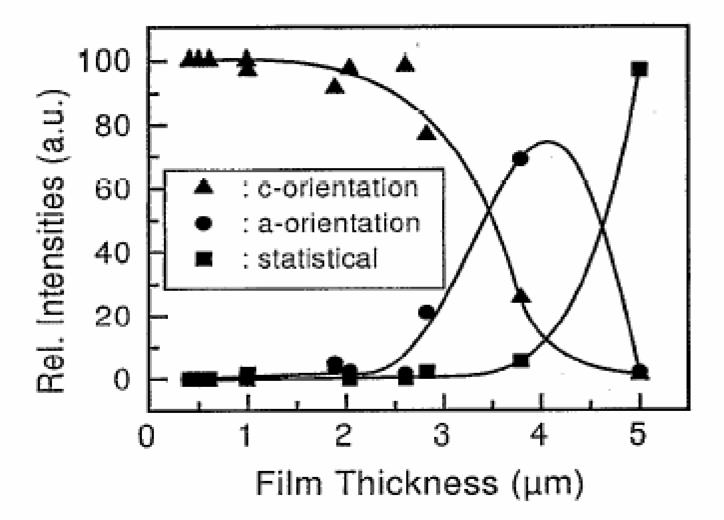
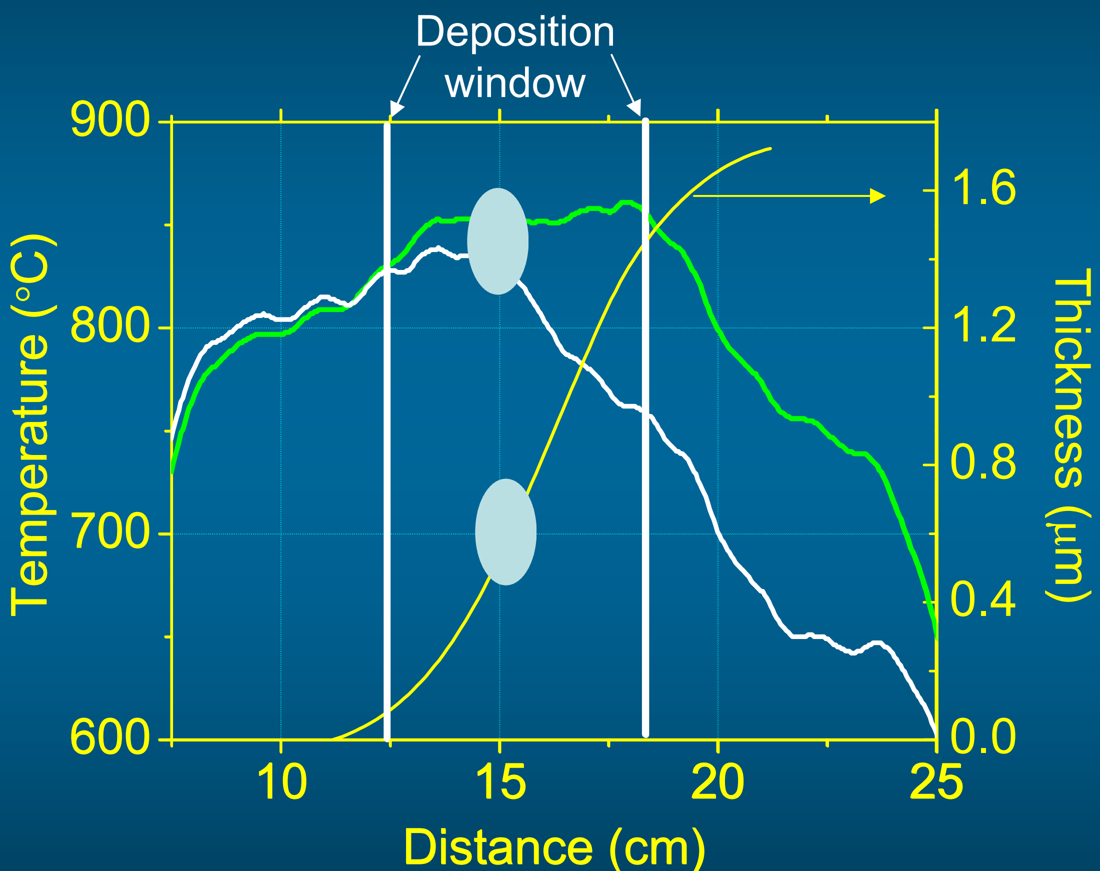
Why implement a zonal heating system?

- Early in high-T_c thin film research, it was found that there was a 'critical thickness' at which film growth sharply transitioned from *c*-axis orientation to *a*-axis orientation
  - Nieh, *et al*. (APL, 1990), Vassenden, *et al*. (Physica C, 1991), Damaske, *et al*. (EUCAS, 1993), Sievers, *et al*. (JAP, 1995)
- This transition is caused by a change in emissivity of the growing film – drop in surface temperature
- By accounting for this, we hope to make higher quality thicker films with higher J_c's



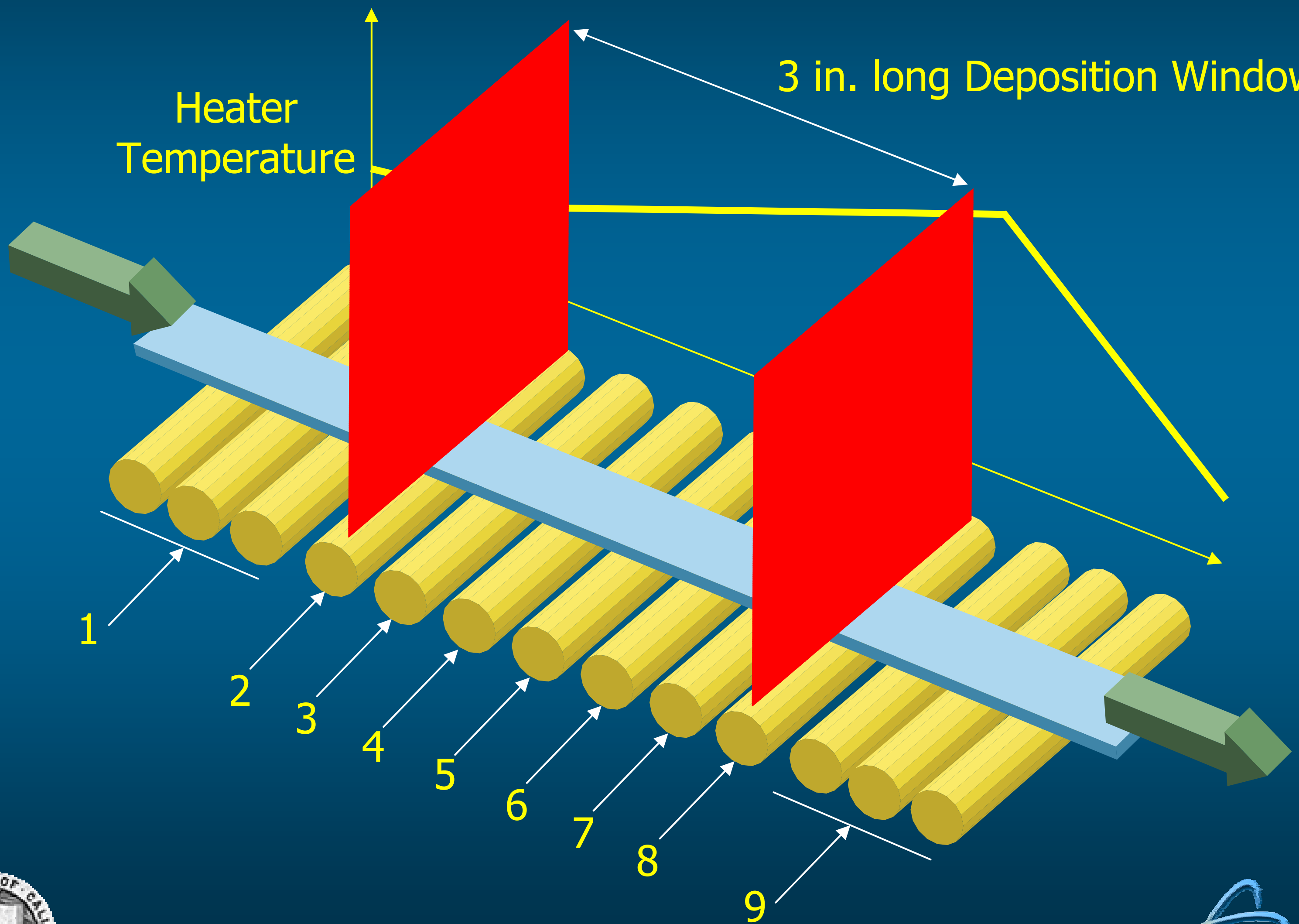
From Sievers, Mattheis, Krebs, and Freyhardt, JAP 78 (9), 1995

What exactly happens to the tape surface temperature as a 1.7 μm film is deposited?

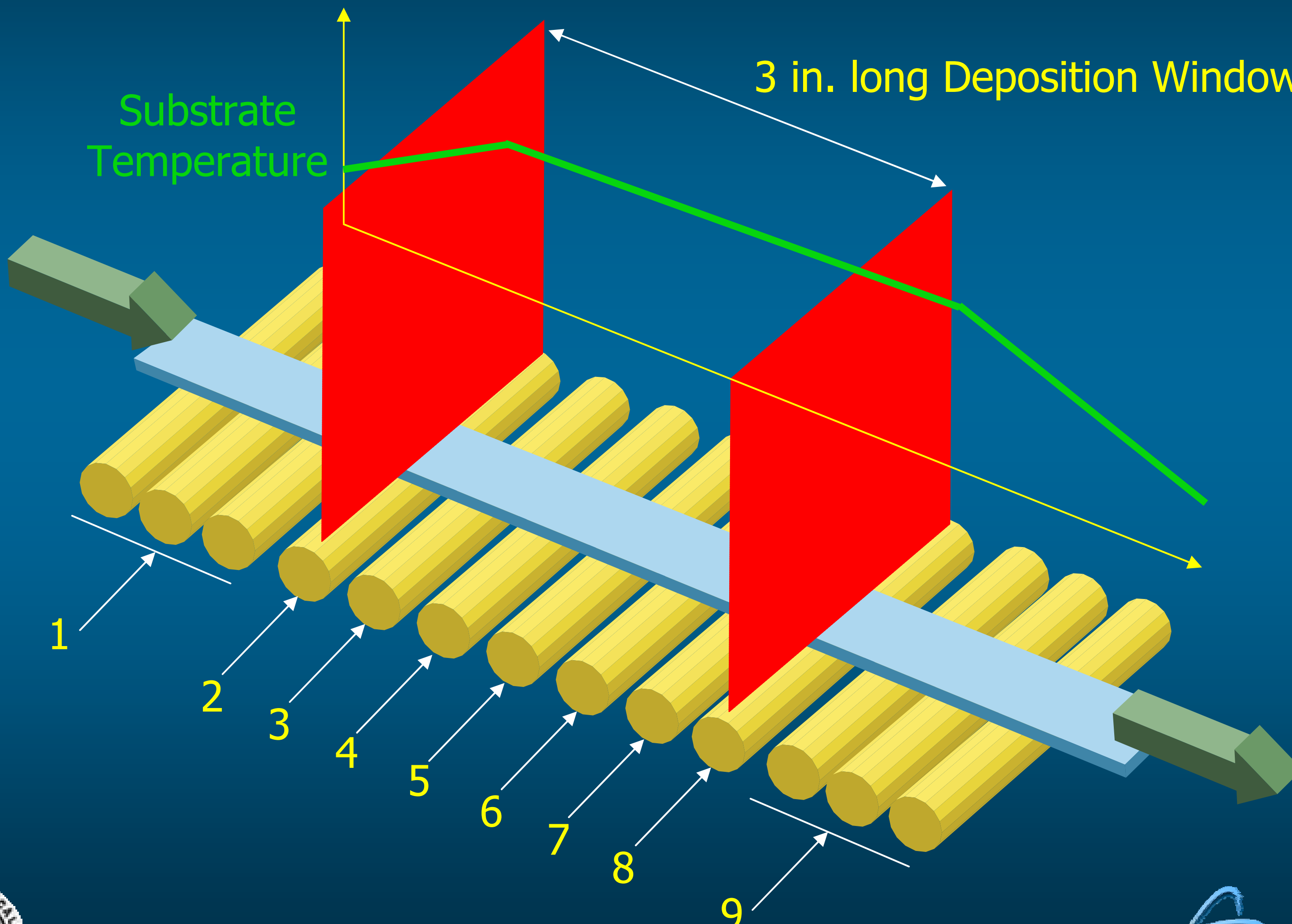


- Trace of the temperature profile with no plume (uncoated)
- Trace of the temperature profile with plume (during coating)
  - Thickness development
- Significant temperature deviation occurs ~0.6 μm

Zonal heater allows for engineered heating profiles: can we account for the change in temperature?

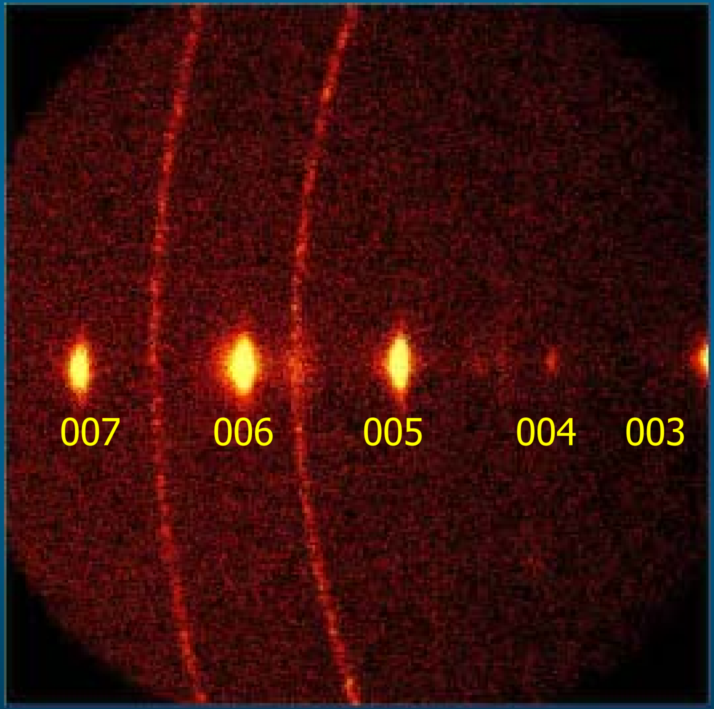


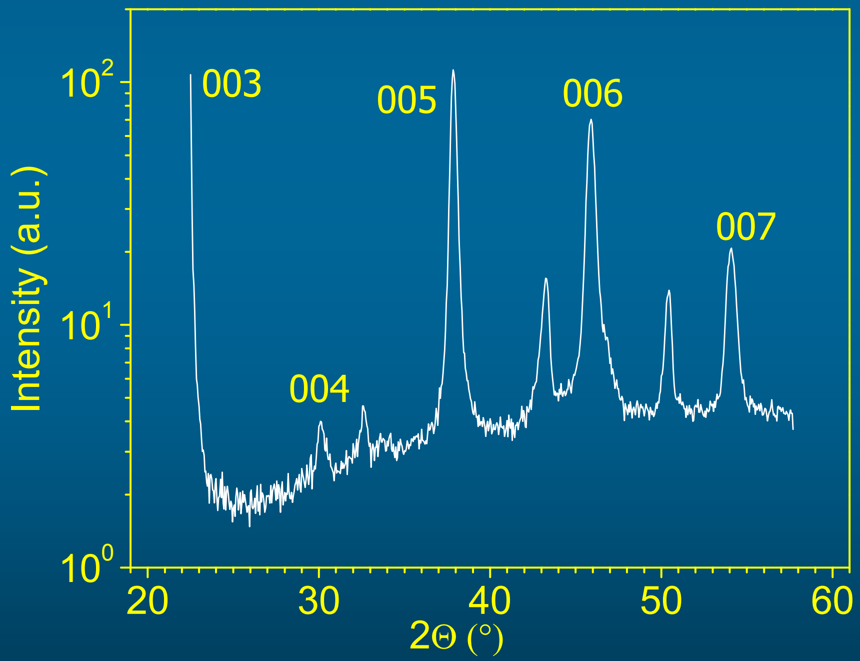
Zonal heater allows for engineered heating profiles: can we account for the change in temperature?



With the engineered heating profile, a 2 μm thick film was deposited

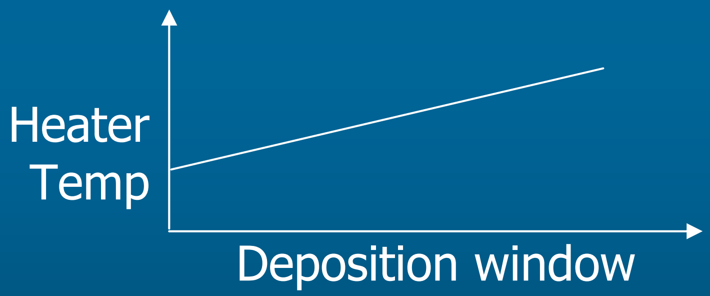
- 200 hz, 1.8 m/hr, FWHM in ϕ = 3.65°



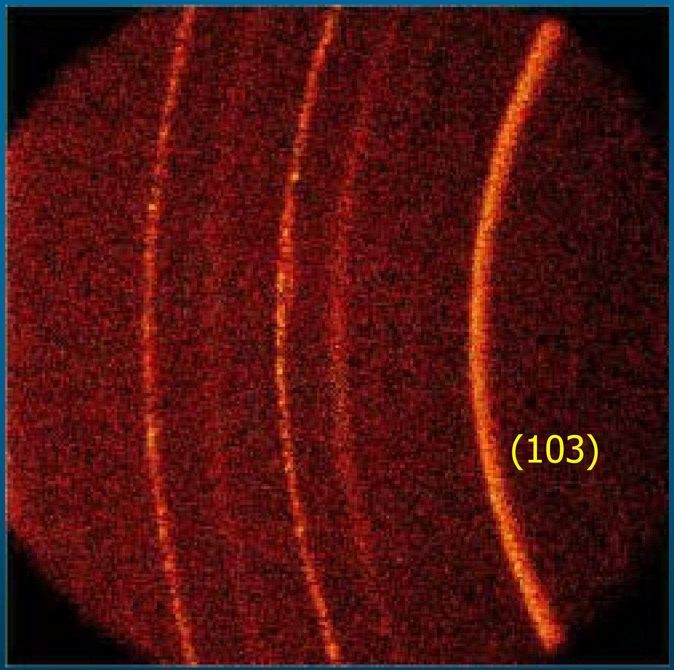


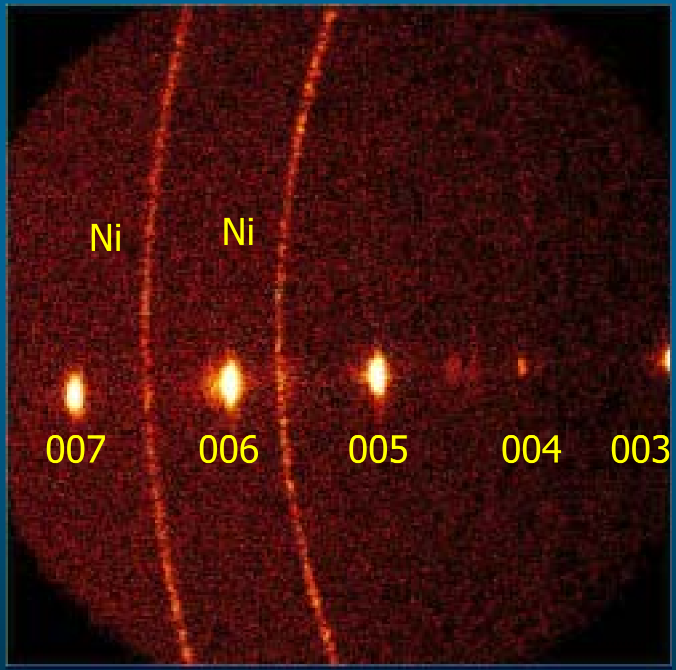
Another advantage to engineered heating profiles is to minimize the thermal budget for high rate depositions

- To demonstrate this, deposit two films under the same conditions aside from the heating profiles:
  - 200 hz, 1.8 m/hr
  - A flat temperature profile
  - An engineered temperature profile

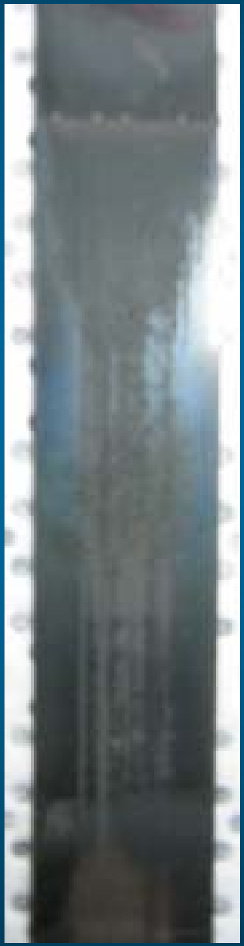


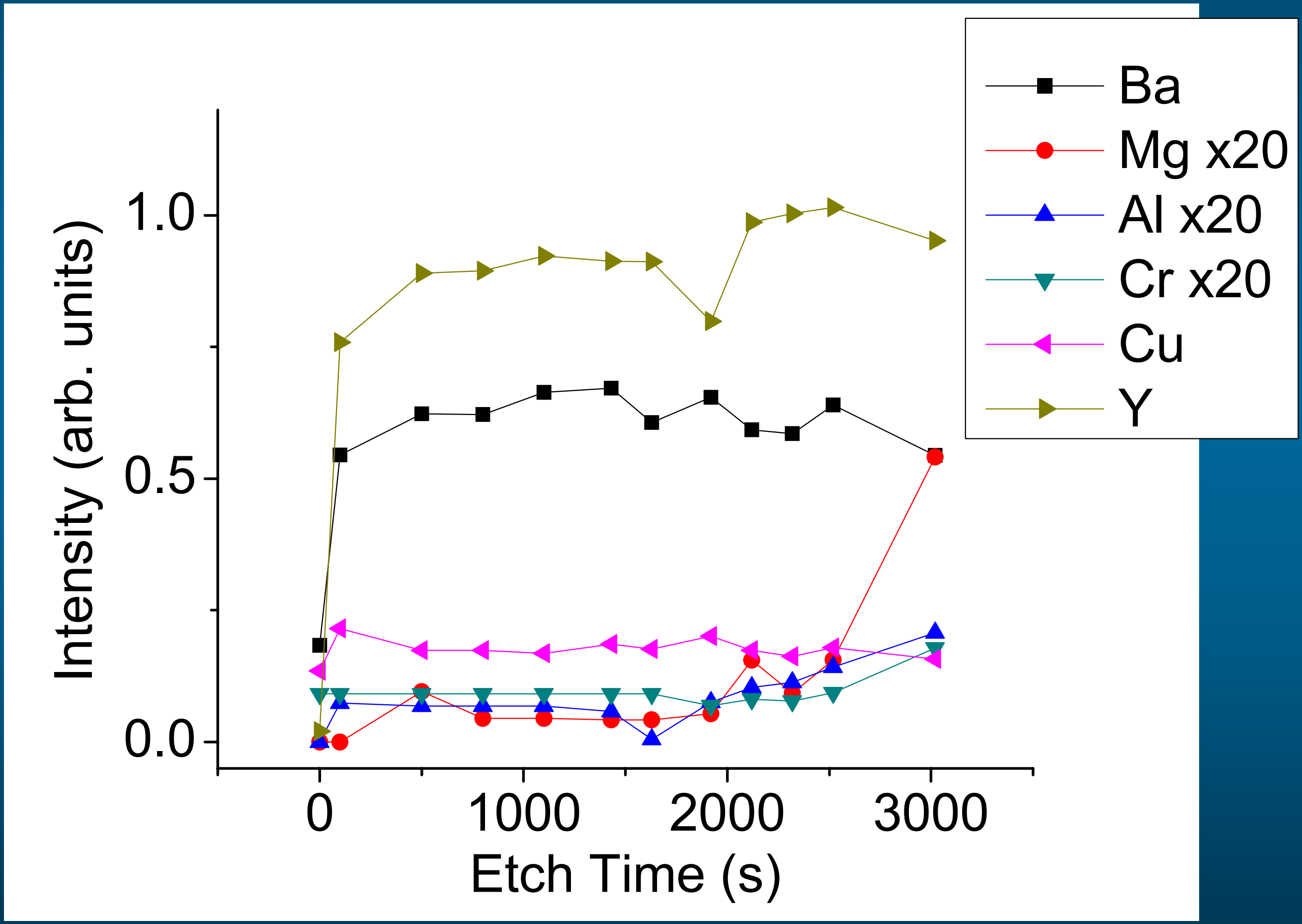
Another advantage to engineered heating profiles is to minimize the thermal budget for high rate depositions



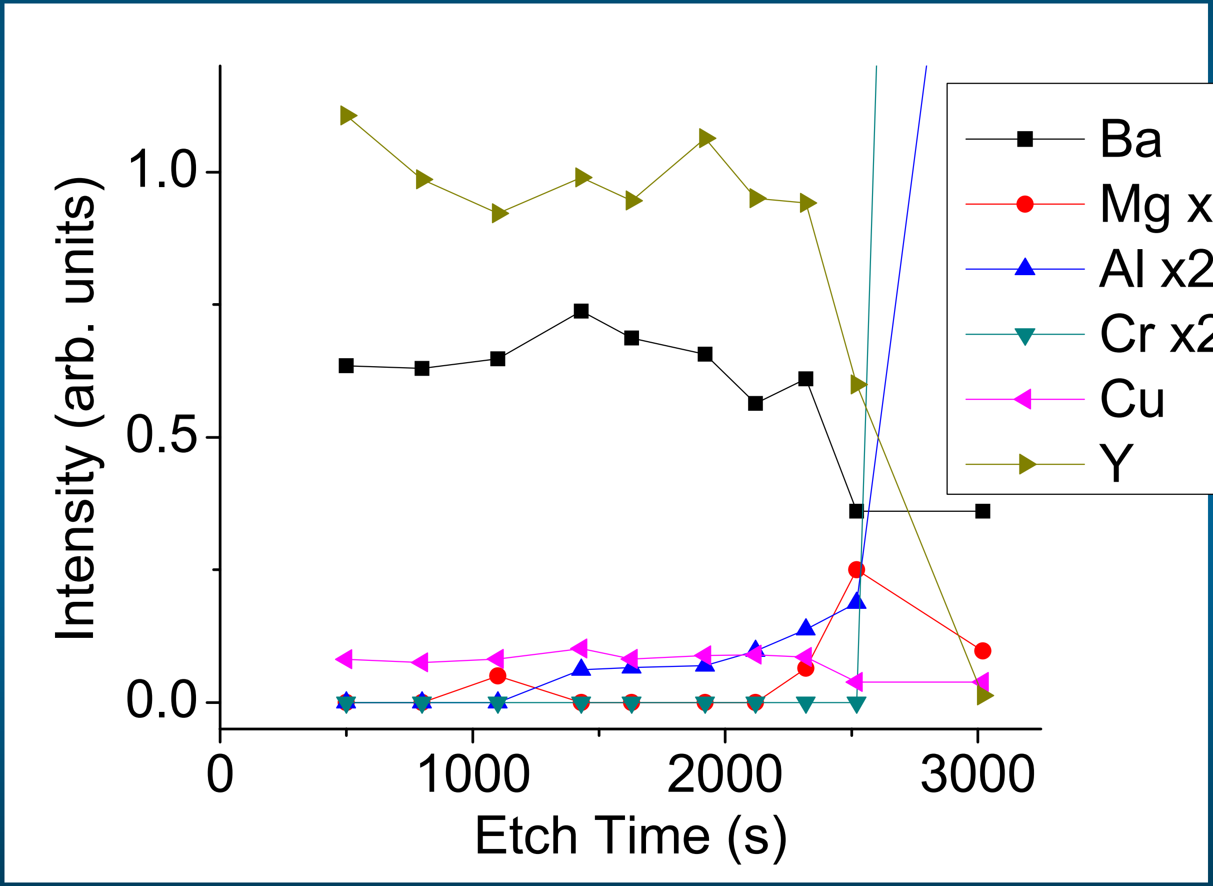


Ion scattering data indicate Cr, Al, and Mg diffuse into the YBCO for a reacted sample

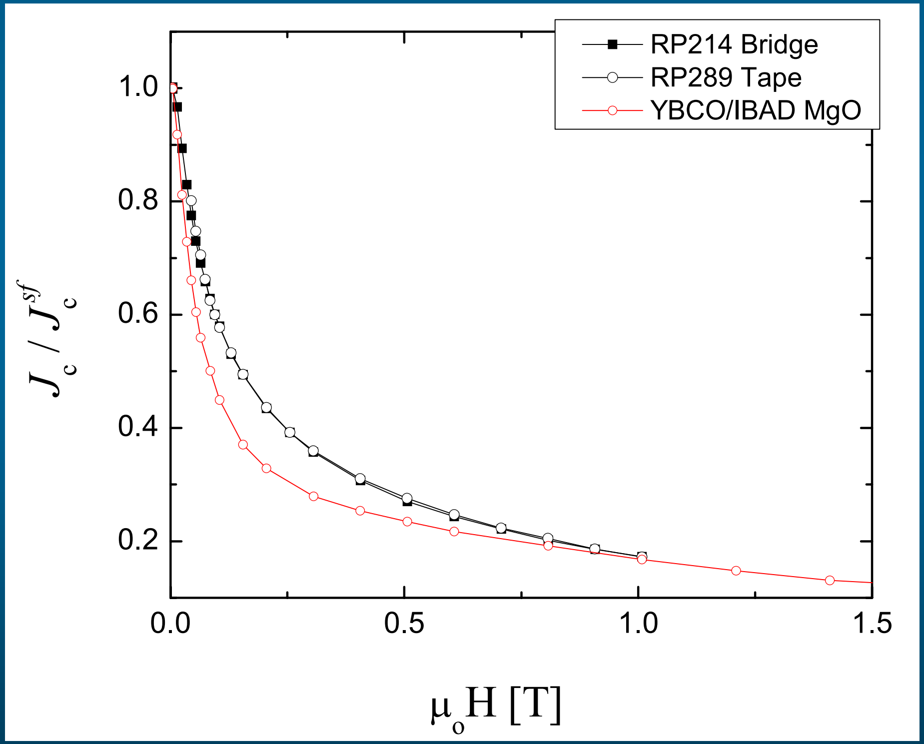




Ion scattering data indicate that Cr does not diffuse into the sample using an engineered heating profile, yet Al is still present



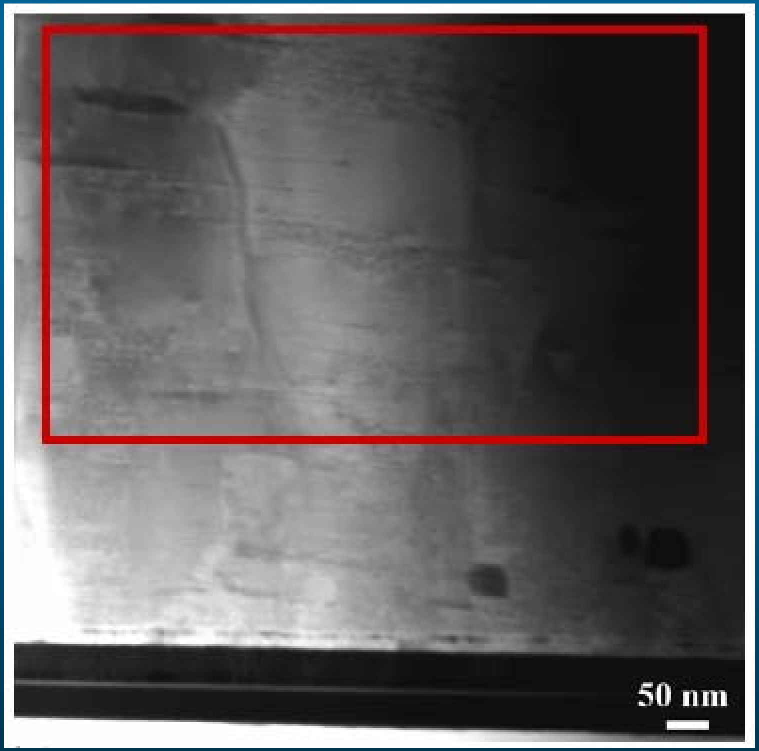
J_c data as a function of magnetic field indicate improved performance as compared to other PLD deposited films

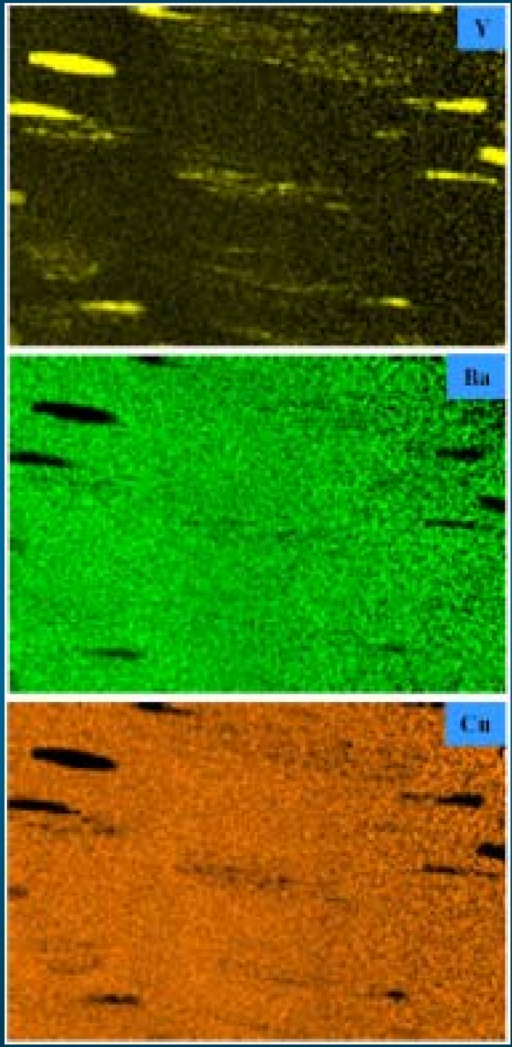


- Especially at lower (< 1 T) fields
  - ~30% improvement at 0.3 T
  - One possible reason is asymmetry in angular dependent data
  - Microstructural data point to another possible reason

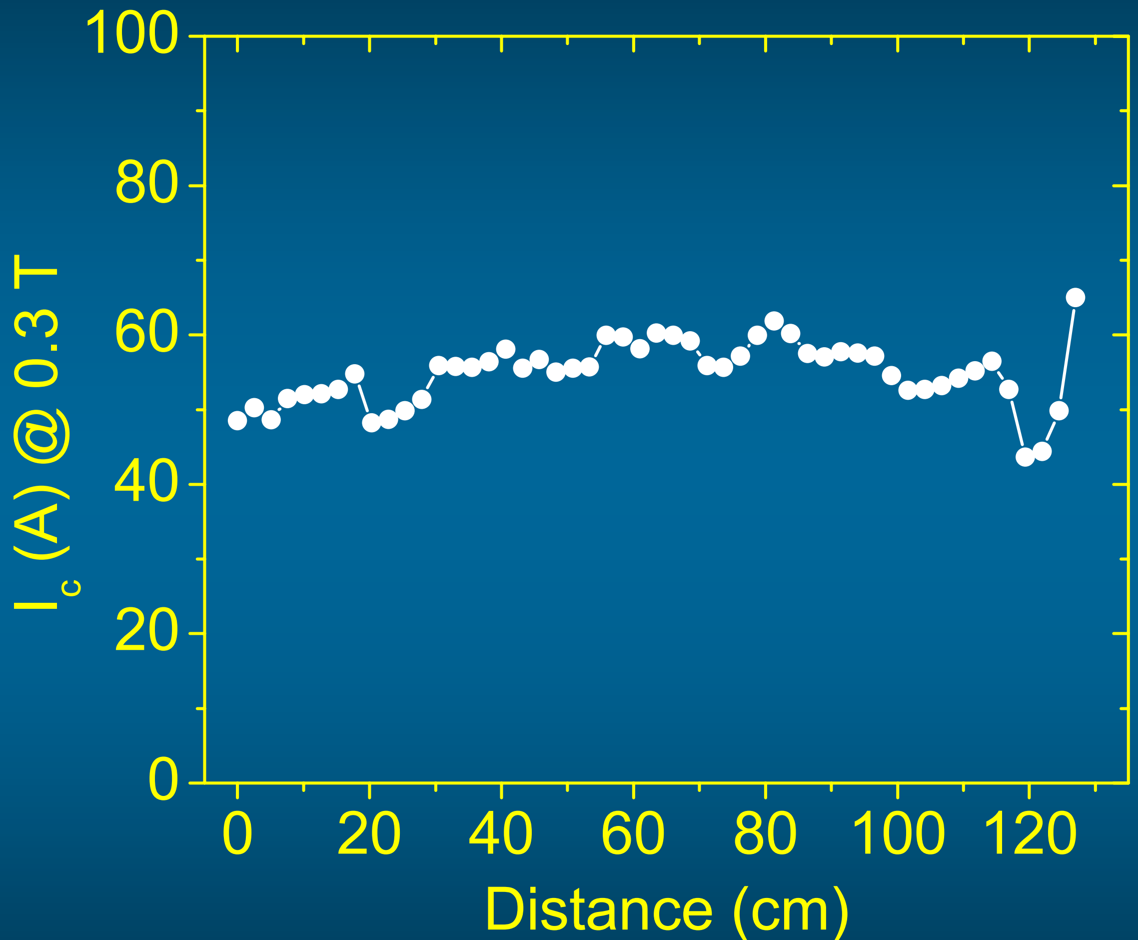
Microstructural data point to a possible reason for the improved in-field data

- Two types of Y_2O_3 precipitates exist
  - 100 nm sized coherent precipitates
  - Particle fields of nm sized precipitates



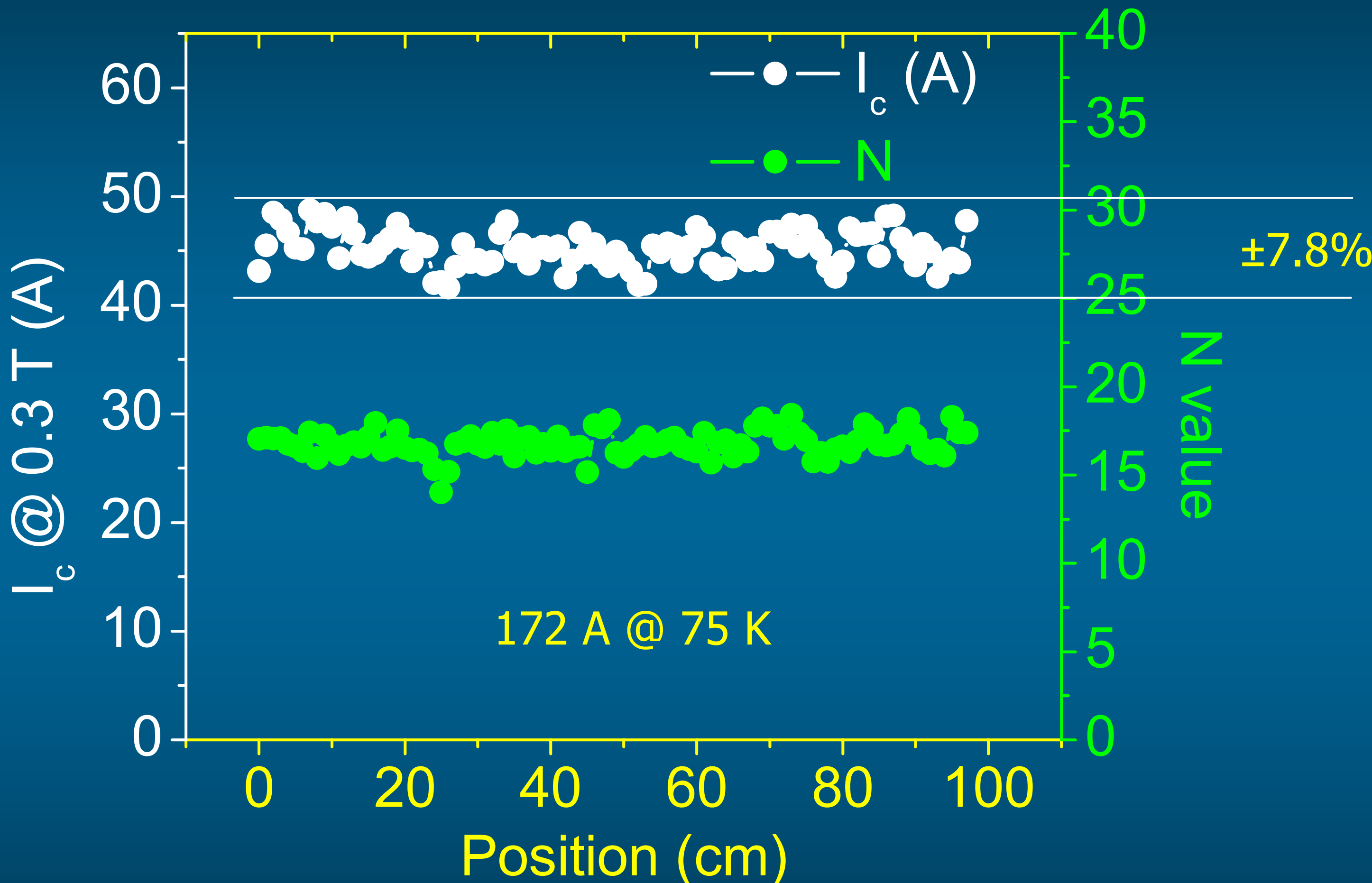


Samples made with new heater show improvement in uniformity, reproducibility, and I_c



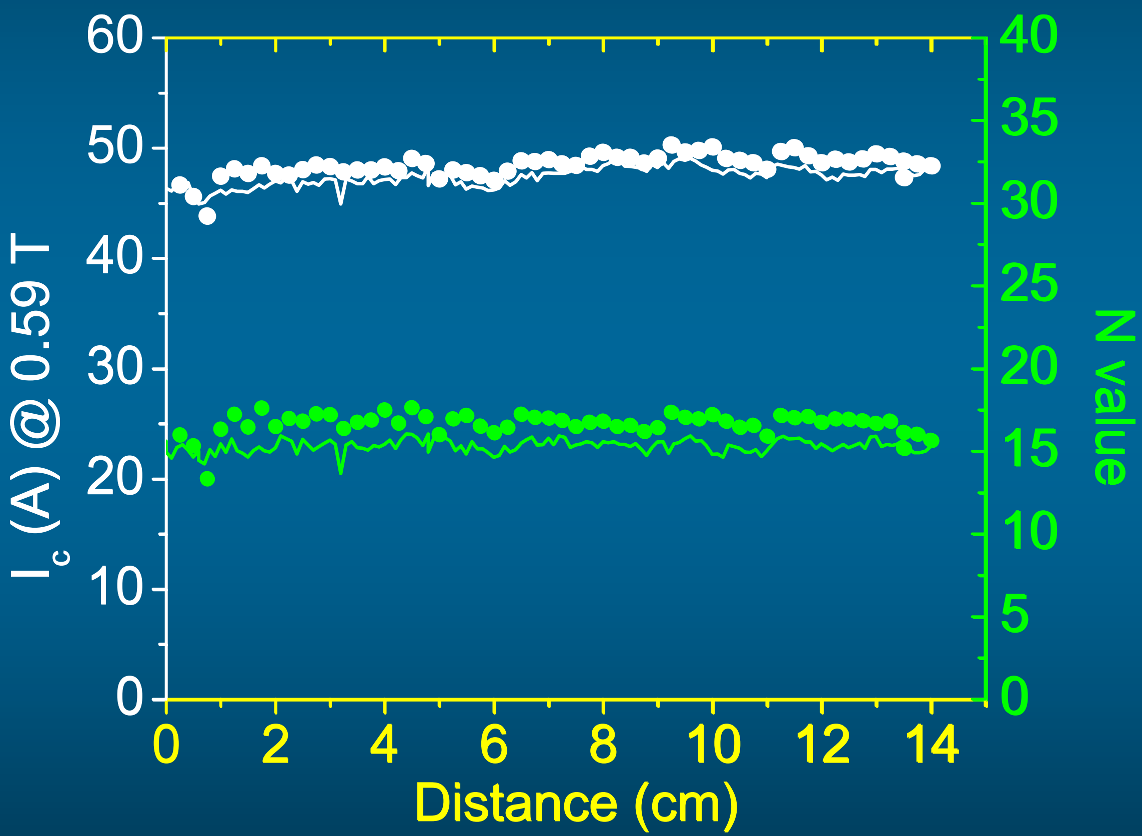
1.1 µm YBCO/50 nm LMO/standard template stack measured at 75 K in 0.3 T

Optimization of engineered heating profile, laser fluence, target-substrate distance, etc. lead to significantly increased length uniformity



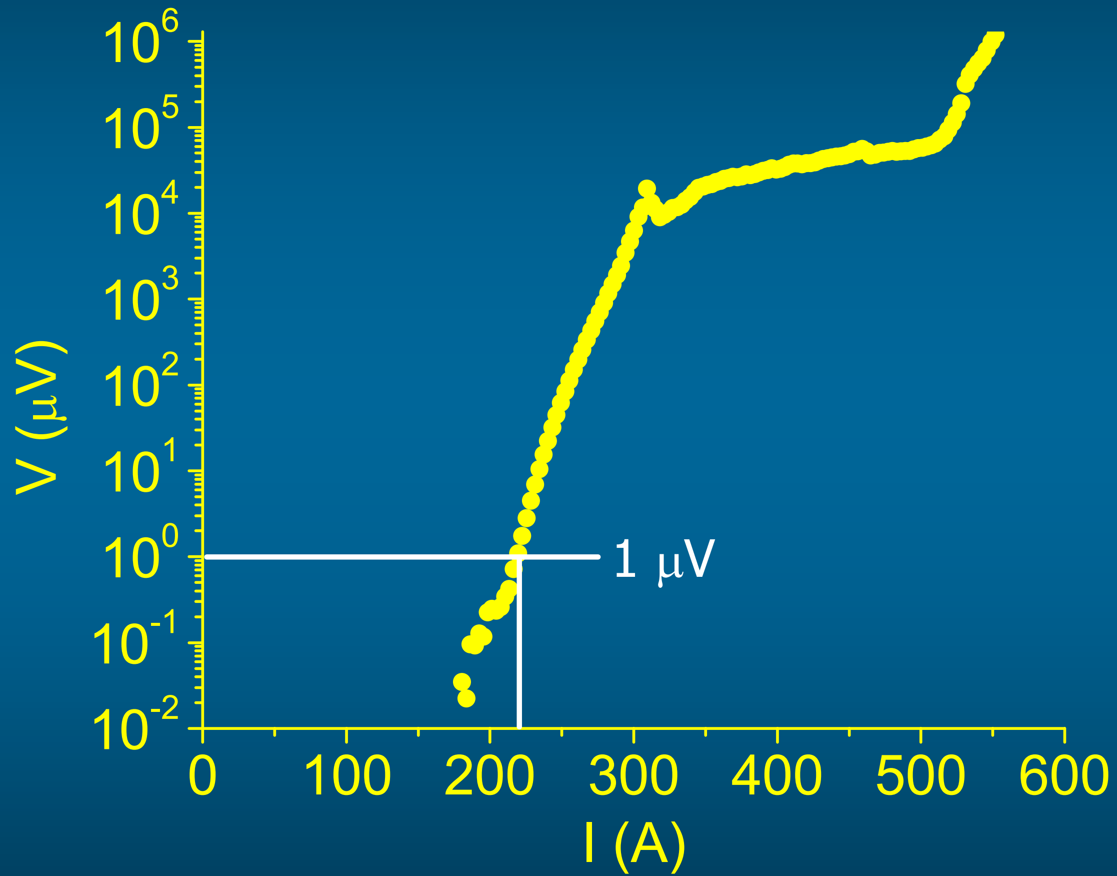
1 µm YBCO/50 nm LMO/standard template stack measured at 75 K in 0.3 T

We've explored the effects of copper plating as a current stabilizer on the performance of our CC's



- Samples plated with a standard Cu sulfate plating bath
  - 50 microns thick on each side
  - Not optimized
- On average, a ~2% drop in I_c is observed after plating
- After plating, uniformity at ±4.5%

Cu plating significantly improves the current carrying ability - up to values approaching 3x I_c

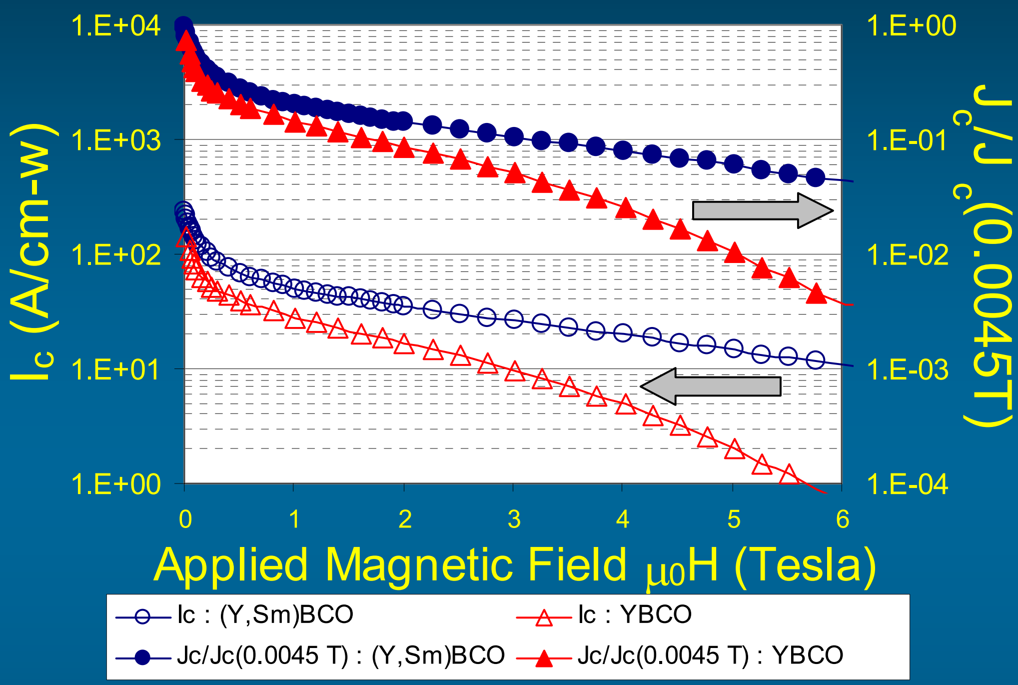


- I_c = 218 A for this sample across 14 cm
- I-V curve run multiple times with no degradation in I_c value

Acknowledgements to Karen Rau and Randy Edwards

Los Alamos

LANL research supported SuperPower in breadth and in depth



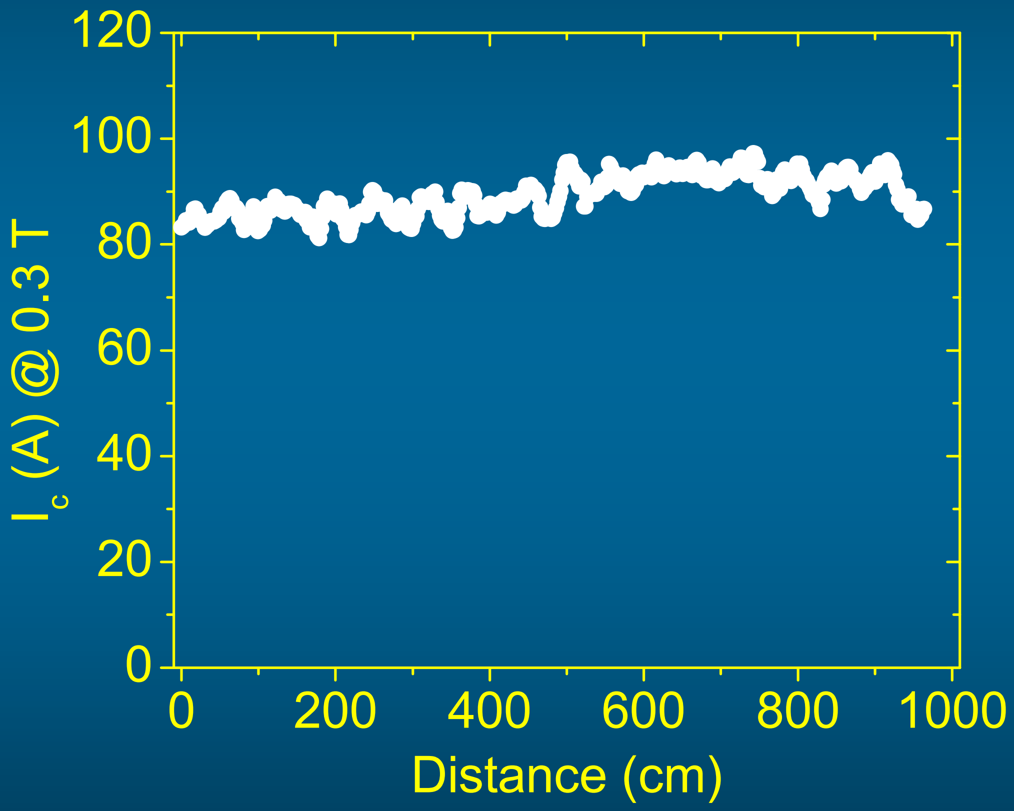
- **<u>Magnetic field dependence studies</u> at LANL enabled SuperPower to develop Sm-substituted compositions that yield improved critical current performance over a large range of magnetic field and essentially all field orientations (Leonardo Civale)**
- **<u>AC loss studies</u> enabled SuperPower to develop striated conductor for low ac losses (Steve Ashworth)**
- **<u>Magnetic Imaging studies</u> enabled SuperPower to improve slitting process (I_c uniformity across tape width) (Fred Muller)**
- **Joint development of <u>reel-to-reel Ic measurement systems</u> for examining I_c uniformity in long tapes (Yates Coulter)**

LANL-SuperPower CRADA spans a wide range of topics and enabled SuperPower to develop an overall superior conductor

SuperPower

Los Alamos

Positional dependence of J_c for a 10 m long MOD/RABiTs tape using the LANL continuous measurement system



- Measurements shown here taken at a 2 cm step size
- Also have measured as small as 1 mm step size

Fundamental challenges for Coated Conductors still remain

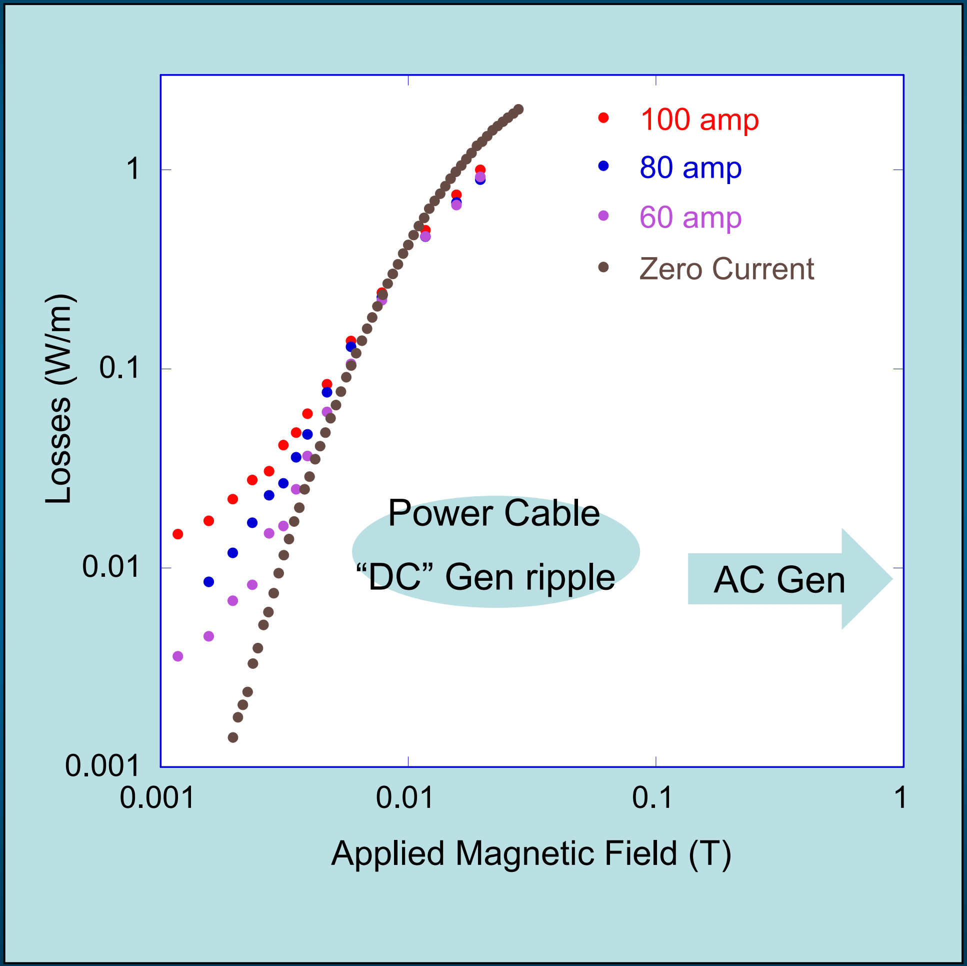
	High I_c	Magnetic field I_c	Mechanical robustness	ac losses
Cables	➽			✘
Motors		➽	✘	✘
Generators		➽	✘	✘
Transformers		➽		✘

➽ significant accomplishments to date

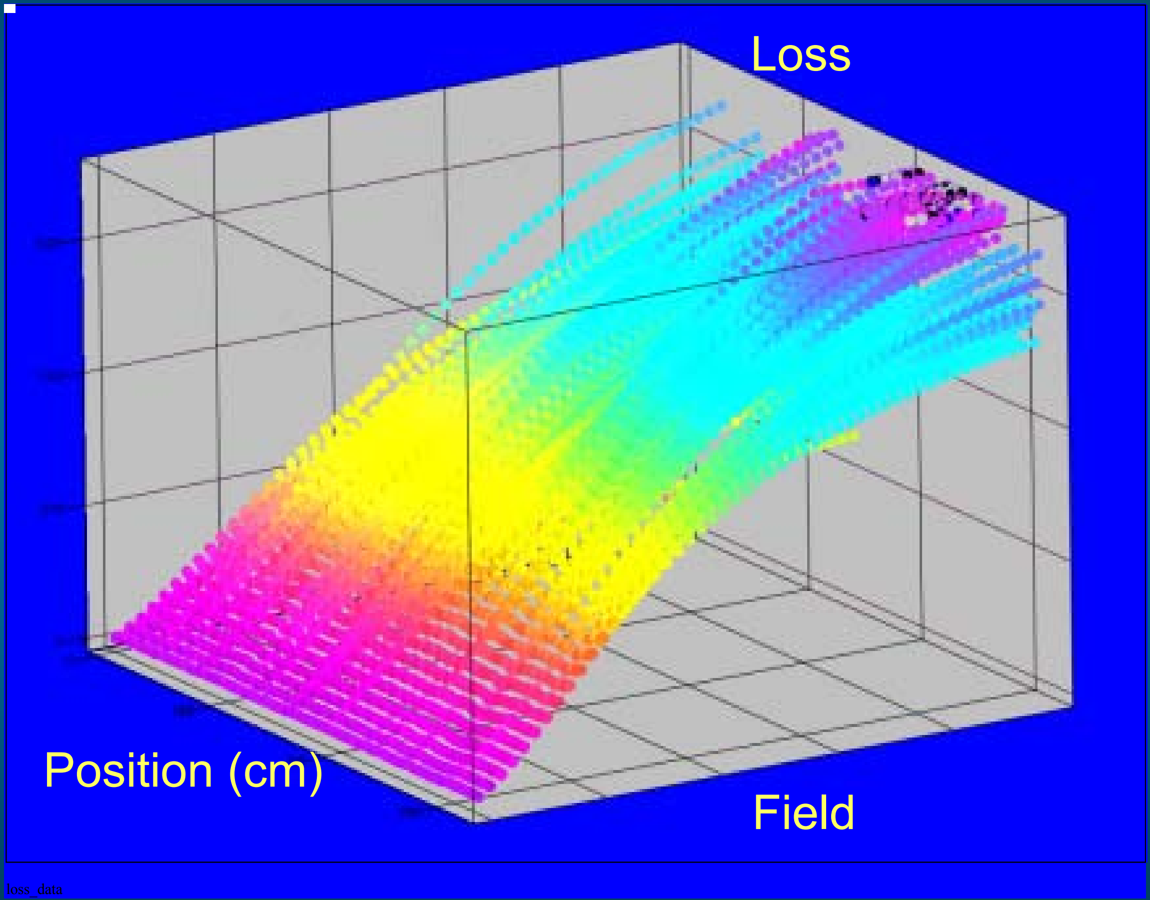
➳ significant progress still needed

ac losses in CC are too high for most applications

- Magnetic field
  - Perpendicular to tape face
  - ac 60 Hz
- Transport current
  - Fraction of I_c (100A)
  - ac 60Hz
- Above 1 mT losses are 'field dominated'
- Losses stimulate quench



New system set up for positional dependence of ac losses over long lengths



- Magnetic ac loss measurement
- Measure every 1 cm
- 5 m tape in 3 hours
- ac loss data can be correlated to J_C across tape

Performance - 2004 Research Park Goals for CC Fabrication & Characterization

- **Goal:** Reduce I_c variation to < 10% on a 2-cm measurement length scale over > 1 m
  - Variation at ± 7.8% over 1 meter

- **Goal:** Fabricate CC > 5 m with I_c > 200 A @ 75 K (J_c > 1 MA/cm^2)
  - 218 A on a 20 cm tape (J_c > 1 MA/cm^2), but have not made a 5 meter long 200 A piece
  - 172 A on 1.3 m
  - Drastically improved J_c's

Performance - 2003 Research Park Goals for CC Fabrication & Characterization

- **Goal:** Provide IBAD-MgO to collaborators in lengths of 10 m with $\Delta\phi$ < 8°; work with Core Program and industry to tailor CC architectures needed for the different YBCO processes
  - Have worked closely with industry and supplied appropriate templates in multiple meter lengths
- **Goal:** Add ion scattering capability to IBAD processing system; utilize for diffusion barrier optimization
  - System is installed and operational; Very satisfied with performance; Have analyzed a variety of samples

Performance - 2004 Research Park Goals for CC Fabrication & Characterization

- **Goal:** Examine the IBAD repair in more detail and optimize HTS layers by maximizing I_c across the repaired regions (> 80% of average I_c)
  - Have shown in IBAD, but not with superconductor deposition; lower priority
- **Goal:** Implement YBCO reactive co-evaporation for CC; goal to make a superconducting 1 m length with 100 A @ 75 K
  - System is installed and operational

Results - 2004

- Electropolishing 3-cm wide tape
- Electropolishing RABiTS
- Ion scattering providing *in situ* data for interdiffusion
- High rate IBAD-MgO at 100 m/hour
- Engineered heater profile for PLD heater
- YBCO 200 A; Core program 1400 A by PLD on short samples
- SP: 116 A over 1.9 m on IBAD-MgO; AMSC: made 198 A MOD-CC on a section of RP IBAD-MgO; MetOx : >0.6 MA/cm^2
- Cu-plating performed successfully; completes wire fabrication

Research Integration - 2004 Research Park

- Provided technology and electropolished tape to industrial partners, Hastelloy and RABiTS substrate
- Provided 10's of meters of IBAD tape to industrial and university partners
- LANL performed structural and electrical characterization on numerous samples from collaborators
- Ongoing collaborations with SuperPower, AMSC, and MetOx with a number of on-site visits
- Ongoing programs with Stanford, U Wisconsin and national laboratories

Rapid transition of IBAD MgO technology to SuperPower

- Apr. 15 : New IBAD facility fully functional at SuperPower
- Apr. 21 - 23 : Paul Arendt & Ray Depaula visited SuperPower to jumpstart IBAD MgO technology transition
- Apr. 26 - June : Joint development of entire structure of IBAD MgO-based conductor at SuperPower

Alumina	Yttria	IBAD MgO	Homo-epi MgO	STO	YBCO	Purpose
SP	SP	LANL	LANL	LANL	LANL	Verified diffusion barrier and nucleation layer
SP	SP	SP	LANL	LANL	LANL	Enabled optimization of IBAD MgO process at SP
SP	SP	SP	SP	SP	LANL	Enabled optimization of sputtering processes at SP

Through close and effective collaboration with LANL, SuperPower was able to fabricate 10 m IBAD MgO tapes with texture of 6 degrees within just 2 months after IBAD system became functional

Systematic & effective collaboration led to demonstration of

- 50 m IBAD MgO tape by SuperPower with texture of 6 degrees and
- 1.86 m long MOCVD tape using IBAD MgO with Ic of 116 A/cm…within 3 months !

AMSC shows high I_c YBCO films on IBAD-MgO through a three way collaboration (LANL-ORNL-AMSC)

LANL: IBAD-MgO template
ORNL: $LaMnO_3$ buffer
AMSC: CeO_2 cap; MOD-YBCO

YBCO/CeO_2/$LaMnO_3$/IBAD MgO

J_c = 2.5 MA/cm^2, sf, 77 K

I_c = 198 A/cm, 0.8 μm

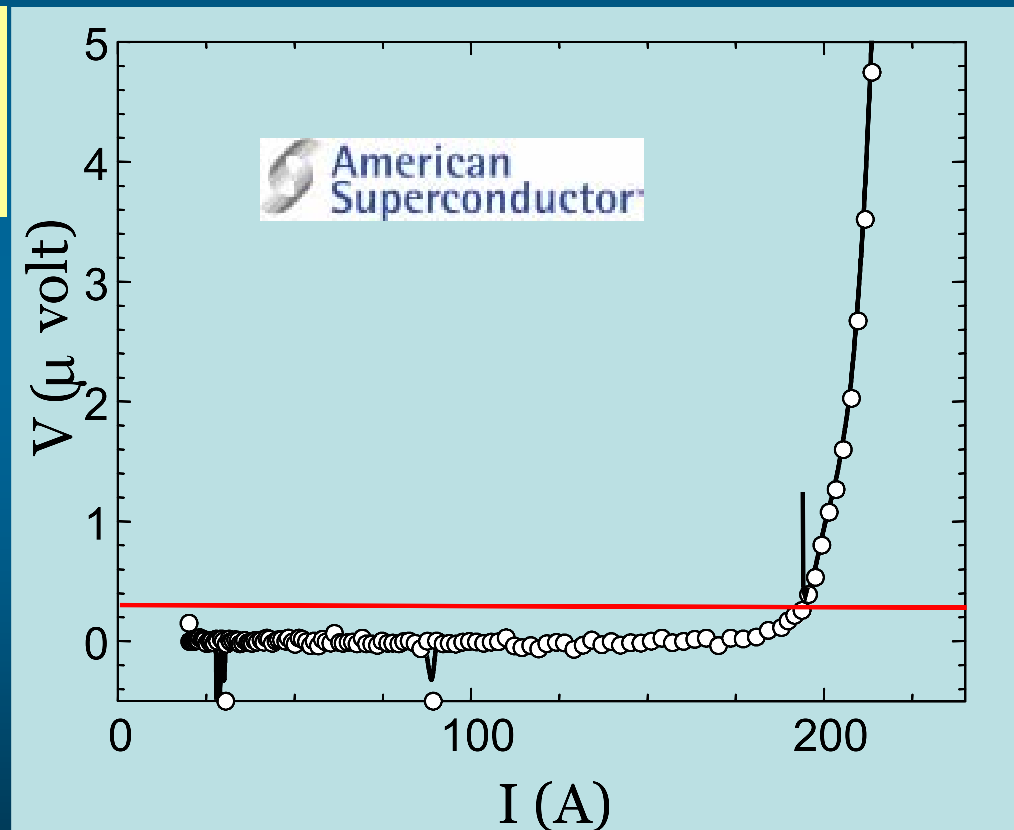
MOD YBCO: $\Delta\phi_{102}$= 3.36°
$\Delta\omega_{006}$= 3.3°, 3.9°

CeO_2

LMO

IBAD MgO

Hastelloy C-276 substrate



Goals for FY 2005 - LANL CC Development

- Add sputtering capability for barrier layer in reel-to-reel system
- Develop robust CC IBAD architectures needed for the different YBCO processes by working with industry
- Reduce I_c variation to < 2% on a 2-cm measurement length over > 1 m; understand the cause of variation
- PLD: Fabricate CC with I_c >500 A @ 75 K (5 m, J_c >1 MA/cm^2)
- Reactive coevaporation: Fabricate CC with I_c > 200 A @ 75 K (5 m, J_c > 1 MA/cm^2)
- Reduce ac losses in the CC wire by one order of magnitude; examine transition from 2D to 3D geometry

Summary

- Electropolishing producing nm-scale smooth tape over 100 m
- IBAD producing 10 m lengths of template tape (~ 6 - 8°)
- IBAD demonstrated at 100 meters/hour
- PLD producing CC meters
- TOF-ISARS surface analysis installed
- Co-evaporation capability installed